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Pesticide Assessment Guidelines, Subdivision N
Chemistry: Environmental Fate

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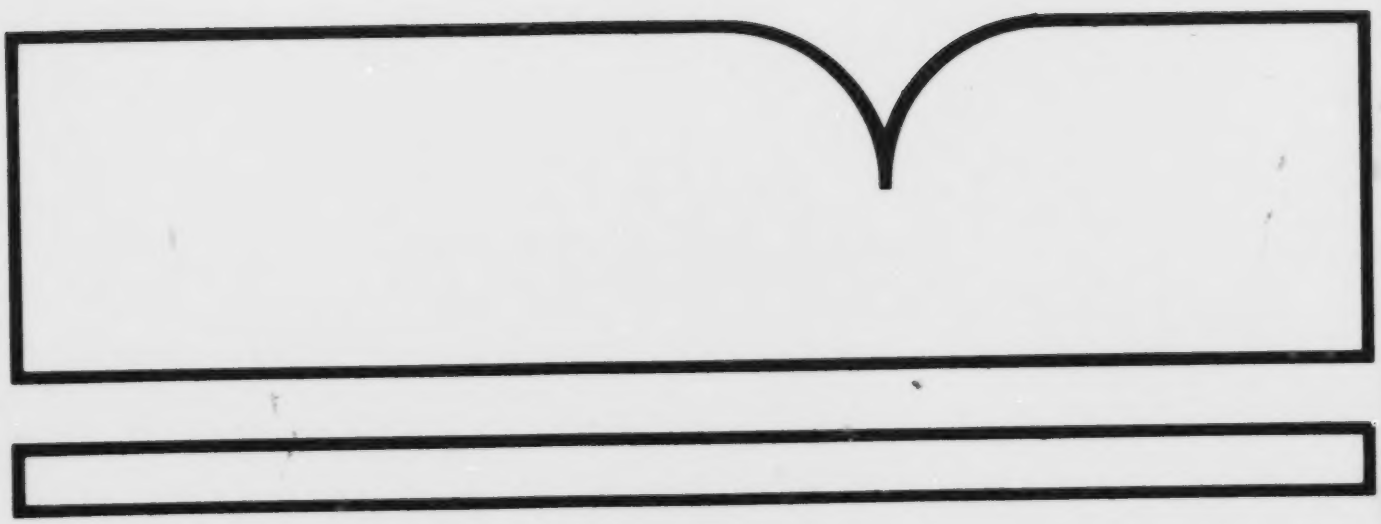

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PESTICIDE ASSESSMENT GUIDELINES

SUBDIVISION N

CHEMISTRY:

ENVIRONMENTAL FATE

by Staff of
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16. Abstract (Limit: 200 words)

This document, Subdivision N, is intended to support 40 CFR 158 requirements for data for the registration of pesticides. Subdivision N describes the Agency's requirements for data on the degradation, metabolism, mobility, dissipation, and accumulation of pesticide chemicals in the environment. These data will be used to identify and assess the potential hazards associated with the uses of a pesticide.

Subdivision N constitutes one volume of a twelve-part FIFRA guideline series published by the National Technical Information service.

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Foreword

Subdivision N describes protocols which may be used to perform environmental fate testing to support the registration of pesticides under the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA). It is a non-regulatory companion to 40 CFR Part 158, Data Requirements for Registration. A public meeting was held in July, 1982 to accept comments on the FIFRA guidelines. Because of the importance of the issues raised in the Subdivision N comments, a review committee comprised of representatives of industry, environmental groups and academia read and approved the Agency revision. This guideline refers to requirements established in 40 CFR Part 158 so that this document can be read as a complete package and so that the protocols may be explained in their proper context.

Subdivision N

ENVIRONMENTAL FATE

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I. ORGANIZATION AND PHILOSOPHY

A. Purpose

The data generated by the environmental fate studies described in these guidelines are used by the Agency, along with other data, to assess the hazards posed by use of a pesticide product, in order to determine whether the product should be registered. In particular, the Agency needs these data to assess:

(1) Direct consequences to man through exposure to pesticide residues remaining after application, either upon reentering treated areas or from consuming inadvertently-contaminated food;

(2) Indirect consequences to man from the presence of widely distributed and persistent pesticide residues in the environment which may result in loss of usable land, water, and wildlife resources; and

(3) Potential environmental exposure of other nontarget organisms such as fish and wildlife to pesticide residues.

Another specific purpose of the environmental fate data requirements is to help registration applicants and the Agency estimate expected environmental concentrations of pesticides in specific habitats where endangered species or other populations at risk are found.

Pesticides can be introduced into the environment by application to water, soil, and air. Following introduction to any of these media, pesticides may be transported from one medium to another by processes that are partly influenced by the chemical characteristics of the pesticide. Transport is also influenced by physical forces in the environment such as meteorological conditions, flow of surface water, location and flow of ground water, tidal action, and topographic characteristics. The physical and chemical characteristics of soil, sediments, particulates, and water also influence the transport and fate of pesticides.

No single test scheme can predict precisely the length of time a pesticide will remain in one particular location, or by what mechanism and at what rate it will be transported, transformed, or degraded. A test sequence at best can provide data which a skilled evaluator can integrate with other information to provide a rough estimate of the concentration of the pesticide and a tentative conclusion about its persistence or rate of disappearance. On the basis of such conclusions, the evaluator can often rule out the

possibility of a pesticide entering certain environments at a concentration that presents a significant risk to man and/or nontarget organisms. On the other hand, the pesticide may enter certain environments at a concentration that would present a significant risk. In these situations, field monitoring studies may provide the information necessary to evaluate the significance of the risk to nontarget organisms.

B. Approach

Proposed rule, 40 CFR Part 158, specifies the kind of data and information that must be submitted to EPA to support the registration of each pesticide under the Federal Insecticide, Fungicide and Rodenticide Act. The Agency intends to promulgate Part 158 as a final rule during 1983. This subdivision provides detailed information that should be included in a test report.

The guidelines also describe circumstances under which an applicant should consult with the Agency before initiating a test. In addition, the references specified in these guidelines provide useful information for designing test protocols and, in some cases, examples for acceptable protocols for conducting the required testing.

The sections of this subdivision of the guidelines have been reorganized since they were proposed as rulemaking in 1978. The following description of the section series, complete with new numbers and the corresponding numbering system as proposed July 10, 1978, will explain this reorganization.

Section 160-1 "General Information" (formerly § 163.62-1 "Purpose: Hazard Evaluation" and § 163.62-3 "Scope and Requirements") describes the purpose of the prescribed environmental fate studies as they relate to environmental hazard assessment. Two tables have been included which summarize the basic Part 158 environmental fate data requirements as they relate to intended use patterns of a pesticide.

Section 160-2 "Definitions" (formerly § 163.62-2) contains definitions of terms used in this subdivision.

Section 160-3 "Use Patterns" (formerly § 163.62-6 "Use Patterns Associated With Data Requirements") categorizes use patterns of pesticides intended for outdoor application.

Section 160-4 "General Test Standards" (formerly § 163.62-4 "Basic Standards for Testing") provides a discussion of the general requirements for test design and test substances applicable to most tests.

Section 160-5 "Reporting and Evaluation of Data" (formerly § 163.62-5 "Reporting of Data") provides reporting requirements that apply to most or all studies.

Section series 161 through 165 (formerly §§ 163.62-7 through 163.62-11) contain specific provisions pertaining to each of the studies required to support the registration of manufacturing-use and end-use products. Each of the five sections series pertains to a single test or related group of tests.

Thus, §§ 161-1 through -4 (formerly § 163.62-7 "Data Requirements: Degradation") relate to physicochemical degradation of pesticides in the environment and describe data requirements for studies of hydrolysis and photodegradation in soil, water, and air. Pesticides introduced into water in the environment may undergo hydrolysis (degradation of the pesticide as a result of interaction with water) and be transformed into new chemicals with properties different from their precursors. The extent to which this process occurs in water can be determined from quantitative data on rate of hydrolysis and half-life determinations. Pesticides introduced into water in the environment can also undergo transformation by photolysis (breakdown by sunlight) into new chemicals with different properties than the original chemical. In certain circumstances, photolysis can also be a major route of degradation of pesticide residues on soil or in the vapor phase. Data on rate of photolysis and halflives establish the importance of photolysis in sunlight as a dominant process in the transformation of pesticides in water. The data generated from both hydrolysis and photolysis studies are then used to determine rate of degradation of the pesticide and identification of pesticide residues which may adversely affect nontarget organisms in the environment.

Sections 162-1 through -4 (formerly §163.62-8 "Data Requirements: Metabolism") describe the Agency's requirements for data related to metabolism which will be used to assess the persistence of a pesticide in a natural environment. These data requirements concern aerobic (in the presence of oxygen) and anaerobic (without oxygen) soil metabolism, and aerobic and anaerobic aquatic metabolism. The data generated from the metabolism studies described in this section are essential to determining the nature and availability of pesticide residues to rotational crops and to aiding in the assessment of environmental hazards related to the persistence of a pesticide in the environment after its disposal.

Sections 163-1 through -3 (formerly § 163.62-9 "Data Requirements: Mobility") describe the data requirements related to pesticide mobility. The data generated from the studies required in §§ 1631 through -3 provide information regarding pesticide transport and are used to assess potential environmental hazards related to contamination of human and animal food, loss of usable land and water resources to man through contamination of water (including

ground water), and habitat loss to wildlife resulting from pesticide residue movement or transport in the environment.

These data requirements pertain to leaching, adsorption/desorption, and volatility of pesticides, and provide information both as to the mode of transport and eventual destination of the pesticide. For example, a pesticide with a high vapor pressure relative to its sorptive tendency (i.e., water solubility, soil absorptivity) is likely to volatilize from the medium where it was applied. A pesticide with a low vapor pressure and relatively high water solubility, in contrast, is more likely to leach through the soil. Volatile chemicals are prime candidates for photolysis, and therefore may cause the presence of photo-oxidants in the air (atmosphere) or otherwise contribute to adverse atmospheric effects. However, nonvolatile chemicals will either bind to the soil or leach, depending on their affinity for particular surfaces and their water solubility. Pesticides that adsorb tightly are generally less subject to environmental transport in the gaseous phase or in solution and may accumulate in the soil. Substances which are not tightly adsorbed, however, can be transported through soils to aquatic systems and to the atmosphere.

Sections 164-1 through -6 (formerly § 163.62-10 "Data Requirements: Field Dissipation") establish requirements for data from terrestrial, aquatic, aquatic impact, and forestry field dissipation studies. A pesticide combination and tank mix dissipation study and a long term field dissipation study may also be required under certain situations. The data generated from the studies described in these sections are used to assess the potential environmental hazards (under actual field use conditions) related to reentry into treated areas, hazards from residues in rotational crops and other food sources, and the loss of land and water resources.

Sections 165-1 through -5 (formerly § 163.62-11 "Data Requirements: ccumulation") describe data requirements for accumulation studies. These studies deal with pesticide residue uptake and accumulation in rotational crops, irrigated crops, and fish. The data generated from these studies are used as part of an assessment of the potential adverse effects of these residues on nontarget organisms. These data also reveal pesticide levels in food supplies that originate from wild sources or from rotational crops. Rotational crop studies are necessary to establish realistic crop rotation restrictions (time from application to time when crops can be rotated) and to determine if tolerances may be needed for residues on such crops. Data from irrigated crop studies are used to determine the amount of pesticide residue taken up by representative crops from irrigation water transported from some other pesticide-treated area. These studies will allow the Agency to establish label restrictions regarding application of pesticides in sites where the residues can transport to irrigated crops.

These data will also provide information that will aid the Agency in establishing any corresponding tolerances that would be needed for residues on such crops. Data from pesticide accumulation studies in fish are used to establish label restrictions (e.g., to prevent applications in certain sites so that there will be minimal residues entering edible fish or shellfish such as catfish or crayfish inhabiting rice fields). These residue data will also be used to determine if any tolerance or action levels are needed for residues in aquatic animals eaten by humans.

Proposed §§ 163.62-12 "Data Requirements: Reentry" and 163.62-13 "Data Requirements: Disposal and Storage" were reserved in the 1978 proposed guidelines. Data requirements for reentry are now treated in Subdivision K of these guidelines; disposal and storage data requirements will be treated in Subdivision P (reserved).

References that appeared in the Appendix to the 1978 proposed guidelines have been carefully scrutinized for adequacy and usefulness. Some were deleted and several new references have been added. All references now include explanatory annotations which better describe the usefulness of each cited reference to the registrant. In the revised guidelines the references have been grouped according to subject matter and relocated at the end of each section.

A listing of current sections and paragraphs in Subdivision N and the equivalent former sections and paragraphs in the environmental chemistry portion of Subpart D in the 1978 proposed guidelines appears in Table 1.

Table 1. Location of current and proposed guidelines.

Subject	Current	Proposed
GENERAL INFORMATION AND REQUIREMENTS		
Purpose and scope of data requirements	160-1(a)	163.62-3
Application of requirements	-1(b)	.....
Approach of subdivision (subpart)	-1(c)	-3(a)
Summary tables	-1(d)	-3(a)
Organization and content of sections	-1(e)	-3(b)
Definitions	160-2	163.62-2
Use patterns	160-3	163.62-6
General	-3(a)	163.62-6
Terrestrial uses	-3(b)	-6(a)
Aquatic uses	-3(c)	-6(b)
Aquatic impact uses	-3(d)	-6(c)
Forestry	-3(e)	-6(d)
General test standards	160-4	163.62-4
Overview	-4(a)	.....
Personnel	-4(b)	-4(a)
Test controls	-4(c)	-4(b)
Test substance	-4(d)	-4(c)
Field studies	-4(e)	-4(d)
Inter-relationship between studies with respect to soil type	-4(f)	.....
Reporting and evaluation of data	160-5	163.62-5
Overview	-5(a)	.....
Nature of submission	-5(b)	-5(a)
Content	-5(c)	-5(b),(c)
Content (data evaluation)	-5(c)(10)	163.62-1(a),(b)
DEGRADATION STUDIES		
General	.....	163.62-7(a)
Hydrolysis studies	161-1	-7(b)
Photodegradation studies in water	-2	-7(c)
Photodegradation studies in soil	-3	-7(c)
Photodegradation studies in air	-4	-7(c)
METABOLISM STUDIES		
General	.....	163.62-8(a)
Aerobic soil metabolism studies	162-1	-8(b)

Table 1. (continued)

Anaerobic soil metabolism studies	-2	-8(c)
Anaerobic aquatic metabolism studies	-3	-8(d)
Aerobic aquatic metabolism studies	-4	-8(e)
Microbial metabolism	.....	-8(f)
General	.....	-8(f)(1)
Effects of microbes on pesticides	.....	-8(f)(2)
Effects of pesticides on microbes	.....	-8(f)(3)
Activated sludge metabolism	.....	-8(g)
MOBILITY STUDIES		
General	.....	163.62-9(a)
Leaching studies	163-1	-9(b)
Laboratory volatility studies	-2	-9(c)
Field volatility studies	-3	-9(c)
Adsorption/desorption	-1	-9(d)
Water dispersal	164-2	-9(e)
DISSIPATION STUDIES		
General	.....	163.62-10(a)
Field dissipation studies for terrestrial uses	164-1	-10(b)
Field dissipation studies for aquatic uses and aquatic impact uses	-2(b)(1)	-10(c)(1),(2)
Specialized aquatic uses	-2(b)(1),(2)	-10(c) 3)
Dissipation studies for forestry uses	-3	-10(d)
Aquatic impact uses	.....	-10(e)
Direct discharge	161-1,-2 162-3,-4 163-1 164-2 165-4,-5	-10(e)(1)
Indirect discharge	161-1	-10(e)(2)
Wastewater treatment	.....	-10(e)(3)
Dissipation studies for combination products and tank mix uses	164-4	-10(f)
Long-term soil dissipation studies	-5	-10(g)
ACCUMULATION STUDIES		
General	.....	163.62-11(a)

Table 1, (continued)

Lab/greenhouse/small plot accumulation studies for rotational crops	165-1	-11(b)
Field accumulation studies on rotational crops	-2	-11(b)
Accumulation studies on irrigated crops	-3	-11(c)
Lab studies of pesticide accumulation in fish	-4	-11(d)
Field accumulation studies in aquatic nontarget organisms	-5	-11(e)
REENTRY	(Subdivision K)	163.62-12
DISPOSAL AND STORAGE	(Subdivision P)	163.62-13

II. RESPONSE TO PUBLIC COMMENT

The Agency received comments from 63 commenters regarding the 1978 proposed environmental chemistry guidelines. In many cases, the commenters provided amended test protocols for consideration by the Agency and provided references or background material. In response to the public comments, the Agency has modified or clarified many sections of the guidelines. For purposes of this discussion, only the more significant and controversial issues reflected by public comments are discussed. Many minor recommendations which do not warrant discussion here were adopted by the Agency.

A. General Information and Requirements.

1. Data requirements for manufacturing-use products. In the Preamble to the 1978 proposed Guidelines, EPA asked for public comment on the question whether the data requirements of this subdivision should be extended to manufacturing-use products. After serious consideration of this issue, the Agency has concluded that extending the data requirements to such pesticides is appropriate. The Agency was influenced by the views of commenters on this issue who generally favored a data submission requirement which makes the basic manufacturer of an active ingredient responsible for providing most of the environmental fate data.

Therefore, a section of 40 CFR Part 158, entitled "Formulators' Exemption" (§ 158.50), requires a registrant of a manufacturing-use product to submit (or cite) any data pertaining to the safety of an active ingredient in its product if the same data are required to support the registration of an end-use product that could legally be produced from the registrant's manufacturing-use products. (An end-use product is a pesticide product bearing label directions for immediate end-use as a pesticide). Section 158.50 also provides that such data must be submitted by an applicant for registration of the end-use product, except that the producer of the end-use product will generally not have to submit or cite data pertaining to registered products which the end-use producer purchases and uses to formulate the end-use product. This decision reflects the Agency's expectation that manufacturing-use product registrants will be the major source of registration data, and that end-use product formulators will, in most cases, need to supply much less data. This decision is consistent with the provisions of, and Congressional intent behind, sec. 3(c)(2)(D) of FIFRA, which provides that:

> No applicant for registration of a pesticide who proposes to purchase a registered pesticide from another producer

in order to formulate such purchased pesticide into an end-use product shall be required to--

(i) submit or cite data pertaining to the safety of such purchased product; or

(ii) offer to pay reasonable compensation otherwise required by [§ 3(c)(1)(D) of FIFRA] for use of any such data.

Implicit in sec. 3(c)(2)(D) is Congress' expectation that it would be the registrant of the manufacturing-use product who would provide significant amounts of data pertaining to the safety of its product. (See, e.g., Sen. Rep. No. 334, 95th Cong., 1st Sess., pp. 8-9.)

Moreover, if data requirements were imposed solely on registrants of end-use products, sec. 3(c)(2)(D) might be read to prevent the Agency from obtaining data on the grounds that the data pertain to the safety of a purchased product.

2. Clarification of Data and Test Substance Requirements

Many public comments revealed that the proposed guidelines created confusion as to which registrants have an obligation to submit data (the "data requirements") and what test substances must be used to generate required data (the "test substance requirements"). For example, several people commented that it was unreasonable to require an aerobic soil metabolism study to be performed with all formulated products. Instead, they argued that EPA should require testing only with the active ingredients in pesticide products. In fact, the proposed environmental fate guidelines did require use of the active ingredient, not the formulated product, as the test substance in aerobic soil metabolism studies. [See proposed § 163.62-8(b) and § 162.8-4(c)(1)(i) of the FIFRA sec. 3 regulations.] Thus, the Agency concluded from this comment and many like it that there was a need to clarify the data requirements and test substance requirements in the environmental fate guidelines.

EPA has taken a number of steps to make these requirements clearer. First, the Agency has included many new definitions and additional explanatory material to clarify which products are covered by a particular use pattern. (See §§ 160-2 and 160-3.) Specifically, EPA has issued a definition for each of the use patterns which serve as the basis for the environmental fate data requirements. In addition, EPA has provided explanatory material in § 160-3 which gives numerous examples of specific uses which are covered by the use pattern definitions.

Second, the Agency has added a section titled "Application status and submittal times" to § 158.30 of 40 CFR § 158 which specifies when data must be submitted for both full registration and conditional registration [FIFRA sec.3(c)(5) and 3(c)(7), respectively].

Third, as discussed above, the Agency has addressed each different study in a separate section of the guidelines. Each of these sections now contains both a "When required" paragraph that states which products must be supported by a particular kind of data and who must submit such data, as well as a "Test substance" paragraph that identifies the substance which must be used in performing the study. Generally, the "When required" paragraphs state that a particular test will be required for all end-use products having a specific use pattern category, and for all manufacturing-use products which may legally be formulated into such end-use products.

Finally, EPA has revised the Table of Use Patterns and Data Requirements that appeared in the proposal. This table, which originally appeared in § 163.62-3, has been divided into two tables that are located in § 160-1 of the current guidelines. The first table covers terrestrial uses and forestry uses, and the second table covers aquatic uses and aquatic impact uses. It must be noted that these tables are intended for quick reference and do not include all data requirements and exemptions from data requirements found in the "When required" paragraphs of the individual sections. For example, Table 1 indicates that a photodegradation study on soil is required to support the registration of end-use products with certain terrestrial uses. The table does not include the provision in the "When required" paragraph for that study which excludes products injected into the soil or incorporated into soil during application. Most of the requirements which are not covered by the tables are listed in an accompanying footnote.

3. Testing a representative end-use product. The Agency seeks to avoid imposing a burden of duplicative testing on applicants for registration. Therefore, where 40 CFR §158 specifies that the test substance shall be a typical end-use product, testing may be performed on a representative formulation. It is not necessary to repeat the test using other similar products. A typical end-use product is defined in §160-2(b)(23) as:

> A pesticide product that is representative of a major formulation category (e.g., emulsifiable concentrate, granular product, wettable powder) and contains the active ingredient of the applicant's product.

The use of a typical end-use product in environmental fate testing is needed for tests which determine the extent of overall pesticide dissipation under actual use conditions. In Subdivision N, all

tests in §164 (dissipation studies) and some in §165 (accumulation studies) are in this category. Moreover, since manufacturing-use products may be formulated into end-use products belonging to several different formulation categories, testing is required with a typical end-use product from each formulation category. Accordingly, the test substance section of these tests now contains a provision which states:

> If the applicant's product is a manufacturing-use product intended for [forestry use], the test substance shall be a product representative of the major formulation category for that general use pattern. (If the manufacturing-use product is usually formulated into end-use products comprising two or more major formulation categories, a separate study must be performed with a typical end-use product for each category.

4. Definitions. In the 1978 proposed guidelines, only one definition -- "material balance" -- was included for the data requirements pertaining to environmental fate studies. In the course of clarifying and revising the subdivision, it became apparent that major terms used throughout the environmental fate sections needed definition. In particular, definitions were needed relating to pesticide use patterns. The four major use categories now employed in the subdivision (terrestrial, forestry, aquatic, and aquatic impact) are now defined, and all of the subcategories (e.g, orchard, aquatic noncrop, etc.) are also defined. See § 160-2. Examples of uses within these categories are provided in § 160-3 (Use Patterns).

5. Personnel requirements. A number of commenters raised objections to proposed § 163.62-4(a), which provided that:

> All testing and evaluation must be done under the direction of personnel who have the education, training and experience to perform the testing and evaluation in accordance with sound scientific experimental procedures.

That section also provided that the Agency may request a resume of the qualifications of personnel performing, supervising, reviewing or evaluating testing. Most commenters objected particularly to the reference to a resume, and stated that if the Agency were to have the opportunity to request resumes, the applicant should have an opportunity to request a resume of the Agency reviewer to determine if the reviewer is qualified to review the data furnished. It was also pointed out by one commenter that the proposed provision was overly broad in that testing is not always performed under the direction of managers and supervisors who have the education, training and experience to perform actual testing.

In response to public comments, the Agency has withdrawn this test standard.

6. Data requirements for small-package, low-toxicity pesticides. Several commenters objected to the imposition of environmental fate data requirements for small-package, low-toxicity pesticide products. They considered the environmental impact of such products to be minimal, and contended that such products should be exempt from these data submission requirements because of the "Agency's obligation to take into account the differences in concept and usage between various classes of pesticides."

The Agency recognizes the position of such commenters, but does not agree. Exclusion of the broad category "small-package, low-toxicity pesticide products" is too general and may preclude the Agency from obtaining information necessary for an assessment of environmental hazards resulting from pesticide products intended for domestic-use applications. For some products, great quantities (on an aggregate use basis) of small-package low-toxicity products used in domestic-use applications can result in adverse environmental effects, even though a single application of such a product would not cause any specific adverse effect beyond intended injury to the target pests. The Agency recognizes that many small-package, lowtoxicity, low-volume products do not pose an environmental hazard even when viewed from an aggregate-use standpoint, and for these, recommends that registration applicants consider submitting requests for waivers of certain data requirements, in accordance with § 158.45 of 40 CFR § 158 [referred to at § 160-1(g) of this subdivision (N)].

However, it should be noted that a single set of acceptable data for each chemical is all that is needed by the Agency. Thus, it is unlikely that individual small package producers will actually be required to conduct such studies, since these studies will ordinarily be conducted and resultant data furnished by a producer of the active ingredient.

B. BENCHMARK CONCEPT.

One commenter expressed the view that the Agency should use benchmark techniques in hazard assessment, and that the data requirements of the guidelines should be drafted with this in mind. The benchmark concept involves using presently available information about chemicals for which data have been generated to predict the behavior of chemicals whose behavior patterns are unknown. The Agency recognizes the advantages of benchmark techniques as a means of hazard assessment, and does not rule out their future use. However, it does not believe that such techniques have been developed to the point where the Agency can rely on such data to predict environmental fate as a substitute for requiring actual data about the chemical for which registration is sought.

C. DEGRADATION STUDIES

1. Hydrolysis. Many commenters suggested revisions in proposed § 163.62-7(b), which set forth proposed requirements for hydrolysis testing. The commenters believe that their proposed revisions to the data requirements would reduce the time and expense involved in performing hydrolysis testing but would still permit the Agency to obtain the data necessary for hazard evaluation.

The proposed version of the guidelines required laboratory hydrolysis studies at two concentrations, two temperatures, and three pH values. Many commenters recommended that the Agency permit hydrolysis testing to be conducted at one concentration and at one temperature. Some of these commenters stated that additional testing at different concentrations or temperatures would be appropriate in the event that initial testing results identified hydrolysis as a major route of degradation of the pesticide.

The Agency agrees with the commenters that testing at one temperature and concentration will yield satisfactory data for hazard analysis. Therefore, the Agency has revised its provisions regarding the test procedure for hydrolysis testing. The guidelines now require "one or more concentrations of the test substance" to be tested, with an upper limit on the permissible concentration of 250 parts per million. The guidelines also permit testing at one temperature, and provide that the temperature of the hydrolysis reaction shall be maintained at 25 $\pm$ 1°C. However, the Agency has retained the requirement that the study be run at pH 5, 7, and 9 because, in its judgment, the three pH's are necessary to span normally-encountered environmental conditions.

2. Soil Photodegradation. Studies of the photodegradation or photolysis of a pesticide on soil surfaces were required by proposed § 163.62-7(c). Numerous commenters objected to the requirement and suggested either deleting or modifying the section. EPA has retained the requirement but has adopted a number of suggestions for its modification. See § 161-3 of this subdivision.

One commenter recommended restricting the requirement for soil photolysis data to those products for which photodegradation is likely to be a significant mechanism of decomposition. The proposed guidelines required soil photolysis data to support the registration of any end-use product with a terrestrial non-crop use, a fruit/nut tree (orchard) crop use, a fieldvegetable crop use, an aquatic use, a forestry use, or direct discharge aquatic impact use.

The Agency agrees with the commenters that this data requirement in the proposed guidelines was overly broad and has added a "When required" paragraph to § 161-3 of the current guidelines which states that soil photolysis data are required to support the registration of only those end-use products with terrestrial crop

uses or forestry uses. The section expressly excludes from the data requirement products with uses which will not result in pesticide residue on the soil surface, i.e., "uses involving injection of the product into the soil or incorporation of the product into the soil upon application . . ."

Most of the comments focused on the test methodology. Some commenters suggested deleting the requirement because of the difficulty and cost of the study or because of the difficulty in interpreting the data. Others argued for its deletion on the grounds that comparable information could be obtained from a "sensitized" water photolysis study. Finally, some commenters proposed specific changes in the test methodology. The change most frequently suggested was that the study be performed on an inert surface rather than on soil.

EPA recognizes that a soil photolysis study requires expensive and sophisticated testing techniques. Nonetheless, the Agency believes that it is appropriate to require the study for products which are likely to leave pesticide residues on the soil surface since soil surface photolysis can be an important factor in the degradation of many pesticides not incorporated into the soil. The Agency has expanded the list of references to include more information on the performance of these studies which should aid those who find the study difficult to perform.

The Agency has considered but does not agree with the suggestion of one commenter that aqueous photolysis studies using water with and without photosensitizers would provide the same information that would be generated by a soil photolysis study. A photosensitizer is a substance which alters the rate or the path of decomposition of a chemical. The commenter's suggestion is apparently based on the assumption that the photodegradation process on soil differs from that in water only in that photosensitizers are present in the former. EPA agrees with the commenter that photolysis in water and soil may differ because of the presence of photosensitizers and that soil is more likely to have such compounds. However, this is not the only way in which soil and water may differ. The Agency anticipates that the amounts and kinds of photoproducts and second order degradation products may differ for studies done in soil and water. For example, a photoproduct formed in water is likely to be hydrolyzed, but the same photoproduct formed in soil could also react with soil organic matter or be metabolized by soil microbes. Consequently, the Agency rejects the suggestion to rely on aqueous photolysis studies.

A number of commenters critized the proposed soil photolysis data requirement because of their belief that EPA would not be able to interpret the data. In particular, EPA would be unable to distinguish between degradation resulting from exposure to sunlight and degradation caused by chemical reactions with the water or organic matter in the soil or by microbial populations in the soil.

The Agency agrees that the study required by the proposed guidelines would not have produced data that could enable it to distinguish among these various degradation mechanisms. In order to do so, the Agency has added a test standard requiring a "dark control," i.e., a requirement that the study be performed both in light and in darkness. [See § 161-3(c)(2)(v).] The differences obtained under the two conditions will indicate to what extent decomposition is attributable to photolysis.

Commenters also suggested that the results of a soil photolysis study are of limited usefulness because the test is performed on only one type of soil. Various types of soils will produce different results due to differences in organic matter content, microbial populations, and photosensitizers. EPA agrees that the differences between soil types will significantly limit the Agency's ability to make quantitative extrapolations of photolysis data from a test soil to another soil type. The Agency, however, does not intend to perform quantitative extrapolations based on these data; rather the Agency's purpose is to obtain an understanding of the importance of photolysis of soil surface pesticide residues and to identify major metabolites formed by this mechanism. The Agency concludes that the test is capable of generating data which satisfy this purpose, and therefore has retained the requirement.

Finally, EPA has considered the frequently-offered suggestion to permit the use of an inert surface such as glass or Teflon instead of soil in this study. Although these studies would be somewhat less expensive, the data generated would not permit the Agency to evaluate the effects of degradation from the combined factors associated with both sunlight and soil, a more natural and more common situation. Therefore, the Agency has rejected the proposed revision.

3. Air photolysis. Most of the public comments on proposed § 163.62-7(c) recommended deleting the requirement for air photolysis studies. While the Agency has not accepted this recommendation, the Agency has revised the proposed provision to provide that the data will be required only on a case-by-case basis and to establish standards for the performance of the test. Several comments noted that the results of an air photolysis study are not needed for non-volatile compounds and compounds that do not vaporize readily. Other comments suggested that the data are needed only to evaluate possible hazards to workers reentering a treated area. EPA agrees with both comments and has added "When required" paragraphs which provide, in § 161-4(b):

> Data from a laboratory photodegradation study in the vapor phase will be required by the Agency on a case-by-case basis to support the

registration of an end-use product with orchard or field-vegetable crop uses that involve potentially significant exposure to workers. Data from such a study will also be required to support the registration of a manufacturing-use product which legally could be used to make such an end-use product. The Agency will make an assessment of what constitutes a significant exposure to workers based on the information required by § 163-2(b)(2).

and in § 163-2(b)(2):

Data from a laboratory volatility study will be required on a case-by-case basis by the Agency to support the registration of each end-use product intended for commercial greenhouse, orchard, or field/vegetable crop uses that involve signficant inhalation exposure to workers. Such studies will also be required to support the registration of each manufacturing-use product which legally could be used to make any end-use product for which laboratory volatility data are required.

(2) The Agency will evaluate the following information provided by the registration applicant to make an assessment of what constitutes a significant inhalation exposure to workers:

(i) Vapor pressure at 25°C and water solubility of the pesticide active ingredient (§§ 64-9 and 64-8 of Subdivision D);

(ii) Soil adsorption coefficient (K_d) of the test substance using the soil from a typical intended application site;

(iii) Soil characteristics, including moisture content, at the intended site of application;

(iv) Method, rate, and intervals of pesticide application;

(v) Temperature, humidity, and air flow rates at the site of application;

(vi) Ventilation sequences or practices for commercial greenhouse applications; and

(vii) Inhalation toxicity of the pesticide (§§ 81-3 and 82-4 of subdivision F).

In accordance with these paragraphs, the Agency will consider both the volatility of the pesticide and the possibility of worker exposure in determining which products will be required to provide air photolysis data.

Other comments stated that requirement for air photolysis data should be deleted because the study is costly and complicated and the results are difficult to interpret. The Agency acknowledges that the test procedures are expensive and difficult to perform and that data are rarely easy to analyze. Nonetheless, the test procedures are reliable, and useful data have been generated from such studies. [See the references in § 161-4(e).] Therefore, when these data are needed to evaluate potentially hazardous exposures, the Agency will require the data.

D. METABOLISM STUDIES

1. Aerobic Soil Metabolism Studies. While most commenters recognized the Agency's need for data from an aerobic soil metabolism study, they had many comments on how such a study should be performed. [See proposed § 163.62-8(b).] The three data requirements in this study which received the most comment were:

- The requirement to use three different types of soil;
- The requirement to continue the study until "ninety percent loss of the pesticide occurs"; and
- The requirement to identify every residue which constitutes more than ten percent of the initial application or 0.01 ppm, whichever is greater.

After reviewing the comments on the requirement to perform the aerobic soil metabolism study on three different soil types, the Agency agrees with the reasons advanced in support of reducing the requirement to one soil type. A number of commenters stated that the variation in results from soil to soil was comparatively small and that there were rarely qualitative differences in the degradation products formed in different types of soil. Thus, the kind of soil seemed to influence only the rate of degradation. They stated, moreover, that rate calculations determined in a laboratory test such as the aerobic soil metabolism study are not highly reliable for predicting degradation rates under field conditions. Accordingly, they concluded that the results of an aerobic soil

metabolism study performed on more than one type of soil would be only marginally useful for the Agency. Many commenters also pointed out that by reducing the number of soil types required from three to one, the Agency could lower the cost of the study. The Agency has been persuaded by these reasons, and has reduced the requirement to only one soil type. See § 162-1 (c)(2)(i) of the current guidelines.

The Agency has also revised the requirements concerning the duration of an aerobic soil metabolism study. The proposed guidelines stated that if the data were used to support the registration of a product with a forestry use, a terrestrial crop use, or a terrestrial non-crop use, the study should last one year or until 90 percent of the active ingredient had degraded and the pattern of formation and decline of metabolites had been established, whichever was shorter. Several commenters stated that the 90 percent loss criterion was inappropriate.

The Agency agrees with the comments. In some cases it will be possible to establish degradation rates for the active ingredient and rates of formation and decline of the degradation products before the active ingredient has degraded 90 percent. In other cases, the active ingredients may degrade very rapidly, so that there will not have been enough time to evaluate the pattern of formation and decline of degradation products by the time that 90 percent of the active ingredient has degraded. In short, the 90 percent degradation figure often may not be relevant to the evaluation of patterns of formation and decline of degradates, one of the Agency's primary concerns in determining the duration of an aerobic soil metabolism study. In order to reflect this reasoning, the Agency has prepared § 162-1 (c)(2)(viii) to read:

> Data must be collected until patterns of decline of the parent compound and patterns of formation and decline of degradation products are established in soil, or for one year, whichever comes first . . .

Finally, the Agency has modified the provisions concerning the level of sensitivity required for the analytical methods used to identify the residues of degradation products remaining in soil. The proposal, § 163.62-8(b), required the identification of residues which constitute ten percent of the initial application level or 0.01 ppm, whichever was greater, and further provided that the experimental dose rate must approximate the field application rate. Public comments identified two problems with this requirement. First, the ten percent criterion was too broad for pesticides applied at high rates (e.g., greater than 5 ppm). If a pesticide is normally applied at 20 ppm (as is the case for some soil fumigants), an applicant would, for example, only have to

identify residues in excess of 2 ppm, 200 times higher than the requirement imposed for other pesticides. Thus, a ten percent criterion would not only deny the Agency access to residue data necessary to make a hazard assessment, but could also indirectly encourage the use of higher application rates. Second, detection of residues which constituted only 0.01 ppm might be difficult.

As a result of the many comments received on this issue, the Agency has revised the criteria for identification of soil residues. The criterion based on 10% of the original application level has been deleted, and the requirement for identification of residues at the 0.01 ppm level is to be taken as a suggested goal to be met or surpassed. See § 162-1(c)(2)(vi). The Agency believes that 0.01 ppm is a valid target level in light of the data received from many registrants and the current literature where this level is either met or surpassed. It is recognized, however, that this level of analysis cannot be achieved for all pesticide chemicals and their degradation products. Therefore, registration applicants will not be penalized for not being able to meet the 0.01 ppm goal due to limitations of the analytical method.

2. Studies of microbial effects. In the 1978 proposed guidelines, the Agency proposed the following sets of data requirements pertaining to microbial effects in relation to pesticides:

§ 163.62-8(f)(2) Effects of microbes on pesticides;

§ 163.62-8(f)(3) Effects of pesticides on microbes; and

§ 163.62-8(g) Activated sludge metabolism.

Subsequent to the proposal, public comments, Agency internal discussion, and the recommendation of the FIFRA Scientific Advisory Panel all focused on the need for the Agency to obtain more definitive information on test procedures and better assessment of the value of test results before promulgating final rules on this subject. The Agency agrees with these arguments, and has not included these data requirements in Subdivision N.

This decision should not be interpreted to suggest that the Agency is no longer interested in microbial effects, nor that their importance in environmental fate of pesticides is not important. It merely reflects agreement among scientists both within and outside the Agency that until useful conclusions can be drawn from properly designed studies, no studies should be required. In this regard, the Agency invites submittal of more suitable and useful test protocols and examples of test results and evaluations that support the protocols.

The microbial effects studies provided in the 1978 guidelines, and the microbial studies in the Subpart J proposal (45 FR 72948)

do not encompass the full scope of microbial effects studies that should be examined if a serious examination of environmental effects involving microorganisms is to be undertaken. When suitable studies can be designed to produce useful, cogent data regarding microbial effects, the Agency is likely to include all such studies within a single, separate subdivision.

E. PESTICIDE RESIDUE MOBILITY STUDIES

1. Soil column leaching studies. Proposed § 163.62-9(b) established data requirements intended to provide the Agency with information about the propensity of a pesticide to leach through soil. The leaching study required was a soil column laboratory study using a minimum of four soils, one of which must be used to study leaching of pesticide residues after the pesticide had been aged in soil under aerobic conditions.

A number of commenters recommended deletion of the requirement for column leaching studies. The consensus of the commenters was that such studies are wasteful of time and resources, because the Agency can obtain sufficient information about the leaching properties of a pesticide from data obtained from adsorption/desorption studies. One commenter gave as an additional reason for deleting the requirement that the Agency had itself recognized a number of problems associated with soil column leaching studies. Another commenter took the opposite view, and recommended that adsorption/desorption data requirements be deleted and the soil column laboratory study requirements be retained.

In response to public comments, the Agency has combined its data requirements pertaining to adsorption/desorption and leaching. The guidelines now provide that a laboratory study is required to provide a quantitative estimate of mobility in soil, but that, with some limitations, the investigator may select which technique to use. A brief discussion of the three techniques follows. Thin-layer chromatography is one laboratory technique which permits the investigator to determine the movement of pesticides in soil coated on glass. A soil column study is another laboratory procedure which provides data on the movement of pesticides in soil. However, the soil used for the study is placed in a vertical column (cylinder), and movement of the pesticide from the top to the bottom is measured. A batch equilibrium study, on the other hand, involves the collection of data on the partitioning of a pesticide between soil and water; in this study, the pesticide is first added to water which is thereafter mixed with soil. The Agency believes that it can give applicants the choice of which techniques to use to measure leachability and still obtain adequate data for the assessment of pesticide mobility.

Therefore, the guidelines now permit data relating to products intended for terrestrial and forest uses to be obtained using soil thin-layer chromatography, a soil column study, or a batch equilibrum (adsorption/desorption) study. A batch equilibrium study must be performed, however, for pesticides intended for aquatic or aquatic impact uses.

2. Effects of Pesticides on Groundwater. Several commenters recommended that the guidelines take into account the potential effects of pesticides on ground water. The Agency agrees and believes that the requirements of Subdivision N will provide the Agency with data that will enable it to ascertain and assess possible ground water contamination by pesticides.

Ground water includes both the aquifers which store water below the earth's surface and other subsurface water sources. It is a valuable resource which has been increasingly used both as a primary and supplementary source of water in many regions throughout the United States. However, since it is a hidden resource, its proper management has often been difficult to achieve. The pollution of ground water is difficult to detect because it usually takes place very slowly. Moreover, once the water is contaminated, the loss of usefulness of the water and the effort to restore the water to a useful state may extend over a long period of time.

The Agency considers that it is probably more cost efficient to prevent the contamination of ground water than to remove pollutants from it. Therefore, as part of the assessment of environmental fate of a pesticide, the Agency will evaluate its potential for ground water contamination. The characteristics of the pesticide that are most pertinent to this inquiry are:

- Leachability;
- Adsorption/desorption characteristics;
- Resistance to chemical, photochemical, and biological degradation;
- Solubility in water; and
- Volatility.

The tests described in Subdivision N provide the Agency with data from which it can assess these characteristics of a pesticide.

It should be noted that, while the data requirements for leaching studies have now been modified, this change is not considered likely to reduce the Agency's ability to assess the risk that a pesticide will contaminate ground water. In the 1978 proposed guidelines, the Agency required that an applicant conduct

both soil thin-layer chromatography testing and a soil column leaching study to measure leachability. As a result of public comment, however, the Agency has been convinced that it can obtain satisfactory data from either of these studies, or from an adsorption/desorption study. Therefore, the guidelines now provide that an applicant submit data from any one of these three studies.

F. DISSIPATION STUDIES: COMBINATION PRODUCTS AND TANK MIXES

A few commenters recommended deleting proposed § 163.62(10(f), which prescribes data requirements for pesticides applied in a combination or tank mix. The commenters questioned whether the Agency has evidence of any adverse effects associated with such combinations, or evidence that a pesticide will behave differently when added to soil at the same time as another pesticide than if it had been applied alone. The Agency has data that indicate that the persistence of a pesticide in soil may increase as a result of its application with another pesticide serially or in a mixture. Based on such information, the Agency believes that the data required by proposed § 163.62-10(f) is useful in hazard evaluation and has retained the data requirement, but only on a case-by-case basis. Refer to PR Notice 82-1, entitled "Revised Policy on Label Claims for Tank Mixing" (January 12, 1982) for a further discussion.

At the suggestion of several commenters, the Agency has amended the provision to require incorporation of the test substance in soil only for those use patterns that require soil incorporation as recommended by label instructions.

G. ACCUMULATION STUDIES

1. Rotational crop studies. Commenters have suggested a number of revisions in proposed § 163.62-11(b), which establishes data requirements for studies of pesticide accumulation in rotational crops. The Agency has substantially revised the requirements for these studies, now in §§ 165-1 and -2, to take their comments into account. The main suggestions and Agency responses are as follows:

(a) Several commenters objected to the requirement for a laboratory or greenhouse study and proposed an amendment to permit a small plot outdoor study. The commenters stated that an outdoor study would more closely approximate growing conditions than would a laboratory study. The Agency has now revised the requirements in

response to these comments to permit the applicant to furnish data from a "confined" (as contrasted to "field") study which can be a laboratory, greenhouse, or outdoor small-plot study.

(b) Several commenters recommended that the guidelines establish a trigger that would determine when field studies would be required. The suggested trigger level of 0.1 ppm of unincorporated extractable residue was proposed by two commenters. A third commenter proposed that a field study should not be required unless there is a single extractable component greater than 0.02 ppm, expressed as parent pesticide compound, present in the raw agricultural commodity. The guidelines, at § 165-2(b)(1) now provide that a field study is required "when significant C^{14} pesticide residues of concern to the Agency are detected in the crops analyzed in the confined accumulation study, § 165-1." Significant residues are defined in § 165-1(d)(1) as "parent compound, closely-related degradates, metabolites and/or their conjugates in the crop, but shall exclude C^{14} activity in the crop associated with that being incorporated into the carbon pool and ultimately into natural plant constituents."

(c) Several commenters suggested that the Agency establish tolerances for residues in rotational crops. The Agency now has a procedure for establishing such tolerances. On January 13, 1981, the Agency issued a policy statement entitled "Tolerance for Pesticides Residues in Rotational and Follow-Up Crops, Meat, Milk, Poultry and Eggs, and for Other Indirect or Inadvertent Residues." Federal Register, Vol. 46 No. 8, p. 3016, Jan. 13, 1981. In that statement, the Agency announced that it will follow the general policy of responding to requests by setting tolerances for pesticides residues resulting from crop rotation or crop replacement practices.

2. Fish accumulation studies. The Agency received numerous comments concerning the proposed data requirements for fish accumulation studies. These commenters addressed four main concerns:

- Commenters proposed that the Agency establish a tier approach to data requirements pertaining to pesticide accumulation in fish.

- Commenters questioned the need for and procedures specified for the static (catfish) accumulation test.

- Commenters expressed concern whether the fish accumulation data requirements in the environmental fate subpart are redundant of or compatible with the fish accumulation data requirements of proposed § 163.72-6 in Subpart E (now § 72-6 of Subdivision E).

- Commenters recommended changes in prescribed test procedures for performing the studies.

The Agency has responded favorably to many of the proposed revisions of this section, and as a result, the fish accumulation test requirements have changed considerably from those previously proposed.

Several commenters proposed that the requirement for a flow-through study should be contingent on whether the parent and/or significant environmental degradation product has octanol/water partition coefficients greater than 1000 and half-lives in water greater than 4 days. The Agency agrees generally with these commenters that a flow-through study need not be performed in every instance, and that there is a high correlation between octanol/water partition coefficient and pesticide accumulation in fish. Nevertheless, the Agency does not believe that testing procedures have been developed that would permit the Agency to use data on octanol/water partition coefficient on a routine basis to determine whether further testing is required. Therefore, the guidelines now provide that the flow-through test requirement may be waived if the applicant can provide evidence that the probability for accumulation is low, based on the physical properties of the pesticide and any other characteristics that bear on its environmental fate. In this way, the Agency has retained the flexibility to require the study for products for which the physical property correlations have been established or for which the use patterns, application rates or environmental fate and transport increase the possibility for accumulation.

A number of comments were received questioning the need for the proposed static "catfish" accumulation study. Commenters expressed concern that no established standard procedures exist for performing the study; that there is a likelihood that the test fish will suffer adverse health effects; that the conditions for the study bear little resemblance to actual environmental conditions; and that several chemical factors decrease the value of the test in assessing accumulation potential. The Agency has carefully studied these comments and has concluded that the major benefit that the study offers, the ability to judge the accumulation potential of pesticide degradation products, is outweighed by the collective shortcomings of the study as currently proposed. More importantly, though, some of these kinds of studies have neither been validated nor verified, and suitable design criteria related to standardization of the techniques are yet to be developed. The Agency has decided, therefore, to delete the static test from the guidelines. The Agency encourages further research in this area, with hope that suitable low-cost reproducible studies that yield the needed information can be developed.

Commenters also suggested changes in test procedures for fish accumulation studies, many of which have been incorporated into the guidelines. Most significantly, the Agency now requires identification of chemical residues only from the two samples with the highest residue levels. Residues below a specified level need not be identified.

Lastly, commenters expressed concern about the relationship between proposed Subparts D and E, both of which required data on accumulation of pesticides in fish.

Subdivision N now requires the following laboratory and field accumulation studies on fish:

(a) Section 165-4 provides that a laboratory flow-through exposure study is required to support the registration of end-use products intended for outdoor uses (except domestic outdoor and greenhouse uses), enduse products intended for aquatic impact uses resulting in direct discharges into the environment, and manufacturing-use products that could legally be used to make such end-use products. However, such data will not normally be required where the applicant can provide evidence that the pesticide and/or its principal degradates will not reach water, will not persist in water, or are unlikely to accumulate in fish.

(b) Section 165-5 provides for a field accumulation study to measure pesticide residues in edible portions of fish under more limited circumstances: if the end-use product is intended for forestry use, aquatic non-crop use, or aquatic impact use that results in direct discharge; if a laboratory accumulation test shows evidence of accumulation, and if no tolerance or action level for fish has been granted.

Subdivision E provides for both laboratory and field accumulation studies (§§ 72-6 and 72-7, respectively), both on a case-by-case basis.

Although Subdivisions E and N both contain data requirements pertaining to the accumulation of pesticides, the Agency believes that it is necessary to retain the requirements in both subdivisions. First, the data requirements in the two subdivisions serve different purposes. Subdivision N contains data requirements designed to obtain information about the hazard from accumulation of pesticides in fish used as edible food sources for humans, and incidentally, other animals that feed on fish or fish products. Subdivision E, on the other hand, contains data requirements intended to provide information on adverse effects of pesticides on fish as nontarget organisms in the environment.

Second, the applicant will not be required to perform duplicate tests to furnish redundant data to satisfy the requirements of both subdivisions. Where one study will satisfy the requirements of both subdivisions, data furnished in response to the requirements of one subdivision may be crossreferenced in the registrant's submission under the other.

The Agency emphasizes that the Subdivision E guidelines require accumulation testing on fish only on a case-by-case basis, with the decision whether to test and what procedure to follow being made after consultation between the registrant and the Agency. Laboratory accumulation testing under § 72-6 of Subdivision E will be required if the active ingredient or its principal degradation products are likely to persist in the aquatic environment, or if they accumulate in the organs and tissues of mammals or avian species. Simulated or actual field testing under § 72-7 will be required if there is evidence that the pesticide is likely to cause adverse effects on fish.

3. Microecosystems studies. The issue was raised that microecosystem testing is not required in the environmental fate guidelines for accumulation studies. The model ecosystem approach, once perfected, could give valuable data as to pesticide mobility, transport, toxicity, distribution, accumulation, and fate. It may be useful, therefore, for the study of current problem areas and pesticides that are currently registered for use. But more importantly, it may eventually be very helpful in providing useful data in future registrations of certain new pesticides as the state of the science of this technique improves.

Scientists are aware, however, that microecosystems are very complex, and data generated by these systems on transformation, transport, and accumulation of pesticide residues are often difficult to interpret. To be useful, several different systems would have to be used. This could result in substantial cost to prospective registrants. More importantly, though, most such systems have neither been validated nor verified, and suitable design criteria related to standardization of the techniques are yet to be developed. The Agency encourages further research in this area, with the hope that suitable low-cost reproducible systems that yield the needed information can be developed.

H. SECTIONS OF 1978 PROPOSAL TO BE ISSUED AS SEPARATE SUBDIVISIONS

1. Reentry. A commenter suggested that the Agency should not defer the issuance of re-entry guidelines but should impose data requirements relating to re-entry at the same time that these and other data requirements are imposed. The commenter expressed concern

that an applicant or registrant might be burdened with duplicative testing requirements if these studies could not be conducted simultaneously. The commenter suggested that reentry data include a determination of hazards due to chronic effects, allergic reactions, and diminished health.

Guidelines have been written by the Agency to establish data requirements to support the establishment of re-entry intervals for pesticides. These requirements are in Subdivision K "Pesticide Assessment Guidelines: Re-entry Protection."

2. Disposal. The 1978 proposal asked for suggestions as to what data should be required relating to pesticide disposal. In response to that request, one commenter suggested that the Agency could require data on incineration parameters and chemical degradation. All comments received in connection with the proposal will be addressed in the development of Subdivision P, "Data to Support Disposal Instructions." This subdivision will take into account the disposal statements in Subdivision H, "Labeling Guidelines for Pesticides and Devices," the proposed guidelines on disposal developed under the Resource Conservation and Recovery Act, and the fact that disposal of pesticide containers must be addressed as well as pesticides.

3. Pesticide Residue Data for Tolerances and Tobacco. Sections in proposed Subpart D dealing with tolerance clearance and with pesticides in tobacco, §§ 163.63-1 and 163.64-1, respectively, will be addressed in a separate subpart entitled Subdivision O, "Pesticide Assessment Guidelines: Residue Chemistry."

I. APPENDIX TO 1978 PROPOSAL

Several commenters pointed out that the Appendix to the 1978 proposed guidelines stated that acceptable test procedures were not available for Items No. 10 (Volatility), 12 (Water Dispersal), 13 (Field Dissipation), 14 (Aquatic Food Crop), 15 (Aquatic NonCrop), 17 (Combination and Tank Mixes), 18 (Long Term Study), and 19 (Accumulation). They consequently recommended that the comparable guidelines sections include only testing for which established protocols are available. Other commenters have indicated to the Agency that many Appendix references were either inaccurate, irrelevant to the subject matter addressed, or out of date and not reflective of the current state of the art in environmental fate studies.

In response to these concerns, the Agency has thoroughly reviewed the references. Those which were judged to be no longer useful for their intended purpose were deleted and replaced by

references to suitable information. In an attempt to make the references more instructive and useful, the Agency has included explanations as to extent of usefulness of each reference.

All references are now located at the ends of the appropriate sections to which they properly correspond, rather than in a single large appendix section.

SUBDIVISION N - CHEMISTRY REQUIREMENTS: ENVIRONMENTAL FATE

Series 160: GENERAL INFORMATION AND REQUIREMENTS

§ 160-1 General information.

(a) Purpose and scope. Pesticides interact with the environment with possible adverse consequences for man and other nontarget species. The purpose of this subdivision is to describe in more detail the Agency's requirements for data concerning the environmental fate of pesticides, which are set forth at 40 CFR § 158.130. These data must be submitted to support the registration of most manufacturing-use products. The Agency will use these data in the identification and assessment of the potential hazards associated with each of the anticipated uses of a pesticide in the different environments in which they are used.

(b) Application of requirements. (1) Application status and compliance. The requirements imposed by 40 CFR § 158.130 and described in this subdivision apply to products already registered as well as those being proposed for registration. The Agency will notify registrants of products already registered, either (occasionally) through the data call-in program, or (routinely) upon development of a registration standard, as to when they must satisfy the data requirements of this subdivision. Refer to § 158.30 of 40 CFR §158 for details of application status and submittal times.

(2) "When required" and "Test substance" requirements. The registration applicant should be careful to distinguish between the "When required" and the "Test substance" paragraph requirements of each section of this subdivision:

(i) The "When required" paragraphs pertain to the circumstances under which data shall be required, and specify the categories of products for which data must be generated to support registration applications. The test data are ordinarily required to support the registration of each end-use product with the prescribed use pattern and each manufacturing-use product used to make such an end-use product.

(ii) The "Test substance" paragraphs relate to the test procedure required to produce acceptable data, and state whether the test substance for a particular study may be the technical chemical, a typical end-use product, or a radioactively-labeled analytical grade chemical.

(3) Testing to meet requirements. Since studies described in this subdivision would ordinarily be conducted by the basic manufacturer, pesticide formulators would not often be expected to conduct such tests themselves to develop data to support the registration of their individual products. They may do so if they wish, but they may also rely on the basic pesticide manufacturer's data already developed. See § 158.50 of 40 CFR § 158 for details of the data submittal exemption for pesticide formulators.

(c) Approach of subpart. (1) This subdivision describes the Agency's requirements for data pertaining to environmental fate of pesticide chemicals. The data requirements are divided into five categories of tests (degradation, metabolism, mobility, field dissipation, and accumulation).

(2) The need for data pertaining to some or all of these tests depends primarily on the pesticidal use pattern. For purposes of this subdivision, pesticides which are used oudoors are categorized by one or more of the following use patterns: terrestrial, aquatic, forestry, and aquatic impact. Applicants should consult the use pattern index (Appendix A of 40 CPR § 158) to determine which use pattern category includes their specific use patterns.

(d) Summary tables. (1) Tables 1 and 2 summarize many of the data requirements set forth in 40 CFR Part 158 and described in this subdivision. Table 1 lists data requirements for terrestrial and forestry use patterns, and Table 2 lists data requirements for aquatic and aquatic impact use patterns. Terms used in these tables have been defined in § 160-2 and described in more detail with examples in § 160-3.

(2) The data requirements for long-term soil dissipation studies are not included in the tables. Also, the following specialized use patterns are not included in the tables: specialized aquatic uses (e.g., antifouling paints on ships and related protective or preservative uses), field volatility, and combination and tank mixes. These uses are covered in the text under the appropriate specific data requirements.

(3) As an aid to identification of testing requirements which may support more than one use pattern, Tables 1 and 2 are presented in a modified bar graph format. Any two or more identified use patterns which are connected by a line on the table have identical environmental fate testing requirements and therefore the data derived from any one test will satisfy the data requirements for all designated use patterns. Where a line does not connect use

patterns on the table, the stringency of the testing requirements differs sufficiently among the identified use patterns so that the data derived from any one test will not satisfy the data requirements for all other designated use patterns. In the case of soil field dissipation data requirements for terrestrial and forest use patterns, however, some degree of substitution of data requirements among identified use patterns is possible. For example, data derived from studies for use patterns (e.g., orchard crop) conducted under more rigorous testing requirements may substitute for or be used to satisfy the data requirements for use patterns (e.g., terrestrial non-crop) that require less rigorous testing requirements, but the reverse is not permissible.

(e) Organization and content of sections. (1) Section 160-2 contains definitions applicable to this subdivision.

(2) Section 160-3 describes the use patterns of pesticides which form the basis for the requirements of this subdivision.

(3) Section 160-4 describes general standards for tests to be performed pursuant to this subdivision.

(4) Section 160-5 contains general requirements for the reporting and evaluation of data.

(5) Section series 161 through 165 provide the specific test standards and reporting requirements for each major data requirement category:

(i) Section series 161 deals with degradation studies;
(ii) Section series 162 deals with metabolism studies;
(iii) Section series 163 deals with mobility studies;
(iv) Section series 164 deals with dissipation studies; and
(v) Section series 165 deals with accumulation studies.

(f) References. (i) At the end of each section are referenced examples of published literature containing acceptable procedures and supplemental background or ancillary material which may be consulted in developing test protocols. To aid the registration applicant in finding appropriate information within the list of publications, the Agency has added a brief annotation following each reference. The annotations point out the usefulness of each publication for development of protocols.

(ii) The Agency recognizes that more than one referenced study may be used in developing a protocol in a given area. In all cases, if the referenced procedure(s) used to develop an alternate protocol contain(s) other information that is inconsistent with

Table I. Summary of Environmental Fate Data Requirements for Terrestrial and Forestry Use Patterns.

	Terrestrial Uses						Test Identical for Aquatic & Aquatic Impact Use Patterns
Data Requirements*	Domestic Outdoor	Green-House	Non-Crop	Orchard Crop	Field and Veg. Crop	Forestry	
Degradation							
Hydrolysis	X	X	X	X	X	X	Yes
Photodegradation							
-water			X	X	X	X	Yes
-soil				X	X	X	
Metabolism							
Aerobic Soil	X	X	X	X	X	X	No
Anaerobic Soil**					X		
Anaerobic Aquatic						X	No
Mobility							
Leaching***	X	X	X	X	X	X	No
Field Dissipation							
Soil****	X		X	X	X		No
Water							
Forest						X	
Accumulation							
Rotational Crop					X		Yes
Irrigated Crop							
Fish			X	X	X	X	Yes
Aquatic Nontarget						X	Yes

* Data requirements cited in § 161-4 (Photodegradation studies in air), § 163-2 (Laboratory volatility studies), § 163-3 (Field volatility studies), § 164-4 (Dissipation studies for combination products and tank mix uses), and § 164-5 (Long-term soil dissipation studies) are not included in this table.

** The anaerobic aquatic metabolism data may be substituted for the anaerobic soil metabolism data but the reverse is not permissible.

*** For domestic outdoor and greenhouse uses a batch equilibrium (adsorption/desorption) study must be conducted.

**** Data derived from studies for use patterns (e.g., field and vegetable crop) conducted under more rigorous testing requirements may substitute for or be used to satisfy the data requirements for use patterns (e.g., orchard crop) that require less rigorous testing requirements, but the reverse is not permissible.

Table 2. Summary of Environmental Fate Data Requirements for Aquatic and Aquatic Impact Use Patterns.

Data Requirements*	Aquatic Uses		Aquatic Impact Uses		Test Identical for Terrestrial and Forestry Use Patterns
	Food Crop	Non-Crop	Direct Discharge	Indirect Discharge	
Degradation					
Hydrolysis	X	X	X	X	Yes
Photodegradation					
-water	X	X	X		Yes
-soil					
Metabolism					
Aerobic Aquatic	X	X	X		No
Anaerobic Aquatic**	X	X	X		
Mobility					
Leaching***	X	X	X		No
Field Dissipation					
Soil (sediment)	X	X			No
Water	X	X	X		
Forest					
Accumulation					
Rotational Crop	X				Yes
Irrigated Crop	X	X			
Fish	X	X	X		Yes
Aquatic Nontarget		X	X		Yes

* Data requirements cited in § 161-4 (Photodegradation studies in air), § 163-2 (Laboratory volatility studies), § 163-3 (Field volatility studies), § 164-4 (Dissipation studies for combination products and tank mix uses), and § 164-5 (Long-term soil dissipation studies) are not included in this table.

** The anaerobic aquatic metabolism data may be substituted for the anaerobic soil metabolism data, but the reverse is not permissible.

*** Mobility studies for aquatic and aquatic impact uses are not applicable for leaching, but are for interpretation of surface runoff mobility and dispersion in an aquatic habitat. Therefore, a batch equilibrium (adsorption/desorption) study must be conducted.

test standards and requirements as specified in the guidelines, then the alternate protocol must be modified to conform with the test requirements in the guidelines.

(iii) In 40 FR 26802 (June 25, 1975, pages 26878-26896), the Agency provided a detailed discussion of many of the tests described in §§ 161-1 through 165-4. This information may be of help to the registrant.

(g) Waivers. Refer to 158.50 of 40 CFR § 158 for details on the policy for waiver of data requirements.

§ 160-2 Definitions.

(a) Terms used in this subdivision shall have the meanings set forth in FIFRA in § 162.3 of the FIFRA Sec. 3 regulations, and in § 60-2 of Subdivision D.

(b) In addition, for the purposes of this subdivision:

(1) The term "agricultural use" means the use of a pesticide product in the production of animals and plants for food, feed, fiber, lumber, flowers, ornamental value, pets, condiments, beverages, chemicals, fuels, smoking and chewing products, and related commodities and purposes.

(2) The term "aquatic crop" means a planting of vegetation and the produce thereof for which all or part of the life cycle involves immersion or suspension of above-ground plant parts in water.

(3) The term "aquatic food crop use" means the use of a pesticide product at any aquatic site for the purpose of controlling pests or providing protection from pests in any aquatic crop grown for the production of human food or domestic animal feed, or for plant regulating purposes in such crops.

(4) The term "aquatic impact use" means use of a pesticide product on water in an enclosed facility or aquatic site that is not accessible to wildlife, birds, fish, or aquatic organisms but from which treated water may subsequently be discharged into natural or outdoor bodies of water accessible to wildlife, birds, fish, or other aquatic organisms.

(5) The term "aquatic noncrop use" means the use of a pesticide product for the purpose of controlling pests or providing protection

from pests in or adjacent to any aquatic site other than that used for production of human food or domestic animal feed, or for plant regulating purposes on such plants.

(6) The term "aquatic use" means use of a pesticide in or adjacent to any outdoor aquatic site readily accessible to wildlife or to aquatic and semiaquatic animals and plants.

(7) The term "direct discharge" of a pesticide means the release, treatment, or application of a pesticide product directly to water at sites within or directly connected to bodies of water to which wild animals, birds, fish, and similar organisms have free access.

(8) The term "domestic outdoor use" means the domestic (non-commercial) application of a pesticide product around (but not in) the home.

(9) The term "field and vegetable crop use" means use of a pesticide product for the purposes of controlling pests or providing protection from pests in any field crop and/or vegetable crop, or for plant regulating purposes in such crops.

(10) The term "forestry use" means use of a pesticide product for the purpose of controlling pests or providing protection from pests in forests, forest tree nurseries, or reforestation sites, or for plant regulating purposes on plants at such sites.

(11) The term "greenhouse use" means use of a pesticide product for the purpose of controlling pests or providing protection from pests in crops grown in commercial greenhouses, or for plant regulating purposes on such plants.

(12) The term "indirect discharge" of a pesticide means release, treatment, or application of a pesticide product to water at sites not directly connected to bodies of water to which wild animals, birds, fish, and similar organisms have free access.

(13) The term "material balance" means an accounting of the quantities of a chemical and its degradates in a defined system based on total radioactivity and/or other recognized analytical methodology.

(14) The term "nonagricultural use" means the use of a pesticide product for all purposes other than those included under the term "agricultural uses."

(15) The term "orchard crop use" means use of a pesticide product for the purpose of controlling pests or providing protection from pests in trees, vines, and shrubs grown or maintained for

production of fruits and nuts, or for plant regulating purposes on such plants.

(16) The term "outdoor use" is generally synonymous with "outdoor application." See sec. 162.3 (cc) of the FIFRA sec. 3 regulations. For the purposes of this subdivision, uses of pesticides in greenhouses, for pulp and paper mill water treatments, and for industrial cooling water treatments are considered outdoor uses.

(17) The term "plant regulatory purposes" means those purposes indicated in sec. 162.3 (ff)(14) of the FIFRA sec. 3 regulations.

(18) The term "serial application" refers to the label-recommended use of a pesticide on a site before or after application of another pesticide to that site, such that the presence of one of the pesticides may affect (usually enhance or complement) the effectiveness and usefulness of the other.

(19) The term "tank mix" refers to the mixture of two or more different pesticide products in a spray tank or other application equipment for the purpose of subsequent application of all products simultaneously.

(20) The term "terrestrial crop" means a planting of vegetation and the produce thereof for which the entire life cycle of the plants involves germination, growth, and maturation in association with anchorage in soil above the water table.

(21) The term "terrestrial noncrop use" means the use of a pesticide product for the purpose of controlling pests or providing protection from pests in any terrestrial vegetation other than that used for production of human food or domestic animal feed, or for plant regulatory purposes on such plants.

(22) The term "terrestrial use" means use of a pesticide in greenhouses or in any or all outdoor land sites except forests and forestry use sites.

(23) The term "typical end-use product" means a pesticide product that is representative of a major formulation category (e.g., emulsifiable concentrate, granular product, wettable powder) and contains the active ingredient of the registration applicant's product.

§ 160-3. Use Patterns.

(a) General. Environmental fate data are required for pesticides intended for outdoor uses. Outdoor uses include most agricultural uses of pesticides and many non-agricultural uses, mostly in

the terrestrial and aquatic non-crop categories. To help specify data requirements to which a pesticide is subject, the Agency has grouped pesticides into the following categories: terrestrial, aquatic, aquatic impact, and forestry uses.

(b) Terrestrial uses. Terrestrial uses include, but are not limited to:

(1) Domestic outdoor uses such as lawn, garden, patio, home swimming pool, and non-commercial greenhouse pesticide uses;

(2) Greenhouse uses, such as for pest control on edible crop plants and on flowering, tropical, and ornamental plants grown in commercial greenhouses. Commercial greenhouse pesticide applications are considered "outdoor uses" since commodities produced in these sites, being of high economic value and requiring intensive pesticide applications for pest control and intensive use of agricultural labor, represent a farm environment with similar - but not identical - pesticide fate characteristics as for many "typical" outdoor uses."

(3) Non-crop uses, such as for vegetation control along utility and highway rights-of-way, at utility substations, in industrial outdoor storage areas, on golf course sandtraps, and at outdoor parking lots.

(4) Orchard crop uses; and

(5) Field and vegetable crop uses.

(c) Aquatic uses. Aquatic uses include, but are not limited to: limited to:

(1) Aquatic food crop uses. These uses include the application of pesticides to areas intended for the production of aquatic or semiaquatic crops such as rice, cranberry, and taro, or for the commercial production of crayfish and catfish; and

(2) Aquatic non-crop uses. These uses generally involve the intentional application of pesticides directly to water to control water pests in areas where food crops are not produced. Uses include applications of products such as larvicides, piscicides, algicides, aquatic weed herbicides, antifouling paints, and molluscicides. Products intended for application to ditchbanks and shorelines are also included in this use pattern category.

(d) Aquatic impact uses. Uses in this category generally involve pesticide applications to water in or within man-made structures (e.g., municipal and industrial wastewater treatments, pulp and paper mill treatments and industrial cooling tower treatments) but which have the potential for adverse consequences which

extend beyond the man-made structures. These consequences may have an impact on organisms in such environments as holding ponds, wastewater treatment facilities, and natural or open aquatic environments. Aquatic impact uses are divided into two categories: direct discharges, and indirect discharges and wastewater treatment.

(1) Direct discharge. This category includes the practice of discharging end-use products directly into the natural aquatic environment in association with their use or the typical method of disposal of pesticide-treated water. Also included are pesticides that are discharged into any man-made aquatic facility where the water is not intercepted by the facility and thus flows directly into natural or open aquatic environments. Since cooling tower water is commonly discharged into lakes, streams, and rivers, most pesticides used to control bacteria or other pests in cooling towers are considered to have direct discharge uses.

(2) Indirect discharges and wastewater treatments. This category includes the practice of applying or supplying pesticides to wastewater treatment facilities where the water is captive, such as for filter fly control. Water in such facilities is generally released only occasionally into natural or open aquatic environments.

(e) Forestry uses. Forestry uses involve pesticide applications to areas that include both terrestrial and aquatic environments (e.g., woods, marshes, streams) consisting principally of forest and reforestation sites.

§ 160-4 General test standards.

(a) Overview. The standards contained in this section apply to all studies in this subdivision unless another section of this subdivision contains a specific standard on the same subject. In such a case, the specific standards in the other section apply to the conduct of that particular study.

(b) Test substance. (1) Data submitted in support of an application for registration shall be derived from tests conducted with the technical (or purer) grade of the active ingredient, a radioactively-labeled analytical grade of the active ingredient, or a typical end-use product, as specified in the test procedure for the particular test.

(2) Although the guidelines permit use of either radiolabeled or non-radiolabeled material as the test substance for some studies, the applicant should be aware that, under almost all circumstances, use of the radiolabeled test substance would generally be the

logical choice for developing satisfactory data. This is particularly true for studies that require a material balance.

(3) The composition of the test substance should be determined, including the names and quantities of known contaminants and impurities, as far as is technically feasible.

(4) The applicant should assign and record lot and sample number of the test substance.

(5) In addition to or in lieu of testing otherwise required by this subdivision, the Administrator may require testing to be conducted with:

(i) An analytically pure grade of an active ingredient;

(ii) The technical grade of an active ingredient;

(iii) An intentionally added inert ingredient of a pesticide formulation;

(iv) A contaminant or impurity of an active or inert ingredient;

(v) A plant or animal metabolite or degradation product of an active or inert ingredient;

(vi) A pesticide formulation; or

(vii) Any additional substance which could act as a synergist to the product for which registration is sought.

(c) Field studies. (1) Location. Field studies should be conducted in areas considered representative of major areas where the pesticide is intended to be used.

(2) Endangered species. Field studies should not be conducted in critical habitats or areas containing or suspected to contain endangered or threatened plants or animals which may be threatened by the test to be conducted.

(d) Inter-relationship among studies with respect to soil type. The same soil type should be used for the accumulation study on rotational crops (§ 165-1), the study of photodegratation on soil surfaces (§ 161-3), the aerobic soil metabolism study (§ 162-1), the anaerobic soil metabolism study (§ 162-2), the leaching study (§ 163-1), and the terrestrial field dissipation study (§ 164-1). The Agency will consider requests for exceptions to this requirement on a case-by-case basis.

§ 160-5 Reporting and evaluation of data.

(a) Overview. This section describes general reporting and evaluation requirements which apply to studies in the subdivision. Each test report submitted under this subdivision should satisfy the reporting requirements of this section, unless a specific section elsewhere in this subdivision directs otherwise.

(b) Submission. Data submitted in response to 40 CFR Part 158 should be provided in a single report to the extent possible. Any data that have been furnished in response to the requirements of another subdivision and that are also required by this subdivision either should be referenced to specific pages in other volumes or should be duplicated and submitted in the volumes containing environmental fate data.

(c) Content. The test report should include a complete and accurate description of test procedures and evaluation of the test results. It should also contain a summary of the data, an analysis of the data, sufficient data for the Agency to verify calculated statistical values, and a statement of conclusions to be drawn from the analysis. The summary should contain sufficient detail to permit the reader to understand the conclusions of the author. In addition to the specific information required by §§ 161-1 through 165-5 of this subdivision, the test report should include the following information:

(1) General information. Each report should contain:

(i) Dates on which study began and ended;

(ii) Name and address of laboratory or institution performing the test;

(iii) Location where the test was performed;

(iv) Names of principal investigators;

(v) Signatures of each of the senior scientific personnel responsible for the study; and

(vi) Certification by the applicant that the report is a complete and unaltered copy of the report provided by the testing facility.

(2) Test method. Each report should contain a statement regarding the test method used, including a full description of the experimental design and procedures.

(3) Test substance. (i) The report should identify the test substance, and should include chemical name and percentage of active ingredient, molecular structure of the active ingredient, and qualitative and quantitative description of the chemical composition, including the results of the analysis conducted in accordance with § 160-4(d)(3), when applicable;

(ii) Manufacturer and lot and sample numbers of the test substances; and

(iii) Properties of the test substance, including physical state, pH, and stability, when not reported for Subdivision D.

(4) Control Values. Due to the wide diversity of pesticide properties, use patterns, and organisms likely to be exposed in the field environment, specific reporting requirements for control values (as to source, sampling regime, and total number submitted) will depend upon the complexity and variability of the environment in which the test is to be conducted.

(5) Test equipment. The report should include a description of the test equipment used, and photographs or detailed descriptions of nonstandard equipment.

(6) Units of measurement. Reporting units should be in the metric system, but the English system may be used, in addition. In no instance should the systems be mixed (e.g., kilograms/acre).

(7) Calculations and tabular, graphic information. Each report should contain the principal mathematical equations used in generating and analyzing data, as well as representative calculations using these equations. When rates of formation and decline of parent compounds or their degradates are reported in any test, data should be expressed as amounts, concentrations, and corresponding percentages. Rate constants, when required, should be reported in conjunction with rate data. Tabular data, as well as graphs for decline curves and soil sorption, should be submitted.

(8) Analysis for and identification of degradation products. Analysis and identification of pesticide residues in field studies is required only for those degradation products that were found to form in the lab studies. This position is taken because the purpose of the lab studies is to identify degradation products and derive kinetics while the purpose of field studies is to derive levels of degradation products as a result of pesticide use under field conditions. Results from the field studies are used in the regulatory process.

(9) Media characteristics. (i) Soils. Characterization of soils utilized in laboratory soil photodegradation, metabolism, and mobility studies, and soils sampled at all field use sites

must be reported, including texture (percent sand, silt, and clay), percent organic matter, moisture content, pH, cation exchange capacity, and bulk density (under field conditions). Soil from foreign sources may be used in those lab studies requiring soil, providing the foreign soil will have the same characteristics as soil in the United States common to the proposed use area. Characteristics to be matched are:

- soil class
- % organic matter
- pH soil
- ratio of soil bacteria to soil fungi to soil actinomycetes

Field studies are to be conducted in the United States.

(ii) Water. In cases of aquatic field tests, characteristics of water obtained from a use site must be reported (e.g., pH, temperature, oxygen content, flow rate, and percent suspended solids).

(10) Data evaluation. Data submitted from studies described in this subdivision will be evaluated by the Agency for relevance to the proposed use, technical adequacy, completeness and overall scientific value, and will be used in assessing the potential occurrence of the following environmental hazards:

Direct hazards to humans;
Direct hazards to fish and wildlife;
Potential for contaminating groundwater;
Potential for magnification in the food chain; and
Potential for uptake by rotational crops.

Series 161: DEGRADATION STUDIES

§ 161-1 Hydrolysis studies.

(a) Purpose. Hydrolysis studies are used to establish the significance of chemical hydrolysis as a route for degradation of a pesticide and to identify, if possible, the hydrolytic products formed which may adversely affect nontarget organisms and may contaminate their food.

(b) When required. Hydrolysis data are required by 40 CFR §158 to support the registration of an end-use product intended for outdoor or aquatic impact use, and to support each application for registration of a manufacturing-use product which may legally be used to formulate such an end-use product. See, specifically, 40 CFR §158.50 and §158.130 to determine whether these data must be submitted. Section II-A of this Subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(c) Test standards. Hydrolysis data submitted in response to 40 CFR § 158.130 should be derived from tests which comply with the general test standards of §160-4 of this subdivisions and all of the following specific test standards:

(1) Test substance. (i) These studies shall be conducted with each active ingredient in the product.

(ii) Where radioisotopic analytical techniques are used (they are preferred), studies shall be conducted with the analytical grade of each active ingredient in the product.

(iii) Where non-radioisotopic analytical techniques are used, studies shall be conducted with the technical or purer grade of each active ingredient in the product.

(2) Test procedures. (i) Laboratory hydrolysis studies should be conducted in darkness.

(ii) One or more concentrations of the test substance should be used for this study. The concentration(s) selected should be within the aqueous solubility range of the pesticide and at a level high enough to define the kinetics of the reaction and also permit isolation and identification of hydrolysis products formed. However, the maximum concentration(s) selected should not exceed 250 ppm. For pesticides of low water solubility (less than 10 ppm), an appropriate solubilizing cosolvent may be added to increase water solubility. However, use of such agents should be avoided whenever possible, and, if used, the concentration of

cosolvent in the final solution should not exceed 1 percent by volume.

(iii) The water used for this study should be free of all live bacteria, and the glassware should be sterilized to minimize the possibility of microbial degradation of the test substance.

(iv) Precautions should be taken during the test to minimize loss of test substance through volatilization.

(v) The temperature of the hydrolysis reaction should be maintained at 25 ± 1°C.

(vi) Hydrolysis experiments should be carried out in solutions buffered at pH's of 5, 7, and 9.

(vii) Results of hydrolysis experiments using high concentrations of buffer should be carefully evaluated to determine whether buffer catalysis effects have occurred.

(viii) Aliquots should be taken at zero time and at sufficient sampling time intervals to define decline of the pesticide and appearance of degradates. The duration of the test need not exceed 30 days.

(d) Reporting and evaluation of data. In addition to data meeting the applicable reporting requirements specified in § 160-5, the test report should contain the following specific information:

(i) The method of adjusting pH.

(ii) Identification of each hydrolysis product produced in greater than 10 percent yield at any point during the course of the study, and material balance and half-life estimates for the parent substance.

(e) References. (1) The following references contain experimental procedures for conducting hydrolysis studies:

(i) Krzeminski, S.F., C.K. Brackett, and J.D. Fisher. 1975. Fate of microbicidal 3-isothiazolone compounds in the environment: modes and rates of dissipation. J. Agr. Food Chem. 23:1060-1068. [The effects of temperature and solution pH on hydrolysis rates are illustrated in this paper.]

(ii) Gomaa, H.M., I.H. Suffet, and S.D. Faust. 1969. Kinetics of hydrolysis of diazinon and diazoxon. Residue Rev. 29:171-190. [This article contains excellent and concise discussions of the kinetics and mechanisms of acid/base catalyzed hydrolysis of an organophosphorus pesticide and its products. It also discusses the effect of temperature on hydrolysis rate, and

illustrates calculation of energies of activation for the hydrolysis reactions. This article is recommended for information on theory and development of rate constants, but the more recent and definitive gas chromatographic/mass spectrographic (GC-MS) technique is recommended for identification of hydrolysis products rather than the methods used here.]

(iii) Under the Toxic Substances Control Act, the Agency has proposed test standards for public review regarding hydrolysis (and certain other studies). These may be found in 44 FR 16240, (March 16, 1979), entitled "Toxic Substances Control: Discussion of Premanufacturing Testing Policy and Technical Issues; Request for Comment."

(iv) [Reserved for: OECD Guidelines for Testing Chemicals. Section 1, Number 111. Hydrolysis as a Function of pH.]

(v) [Reserved]

(2) Information on GC-MS for identification of pesticides and products can be found in:

(i) Ryan, J.F. 1977. Residue Analysis Applications of Mass Spectrometry. Pp. 1-49 in Analytical Methods for Pesticides and Plant Growth Regulators. IX. Spectroscopic Methods of Analysis. G. Zweig and J. Sherma (eds). Academic Press, Inc. N. Y.

(ii) Wolfe, N.L., R.G. Zepp, G.L. Baughman, R.C. Fincher, and J.A. Gordon. 1976. Chemical and Photochemical Transformation of Selected Pesticides in Aquatic Systems. Ecological Research Series. USEPA 600/3-76-067. 141 pp. [This publication contains reviews and experiments on the photolysis of various pesticides in water. Extensive information is presented that may be useful for the design of experiments and evaluation of data.]

§ 161-2 Photodegradation studies in water.

(a) Purpose. Pesticides introduced into aqueous systems in the environment can undergo photolytic transformation by sunlight. Data on rates of photolysis and half-lives are needed to establish the importance of this transformation process and the persistence characteristics of photoproducts formed.

(b) When required. Data from a photodegradation study in water are required by 40 CFR §158 to support the registration of an end-use product intended for any terrestrial use (except greenhouse and domestic outdoor use), aquatic use, or forestry use, or for any aquatic impact uses involving direct discharges of treated

water into outdoor aquatic sites. Data from such a study are also required to support each application for registration of a manufacturing-use product which may legally be used to formulate such an end-use product. See, specifically, 40 CFR § 158.50 and §158.130 to determine whether these data must be submitted. Section II-A of this subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(c) Test standards. Photodegradation data submitted in response to CFR §158.130 should be derived from tests which comply with the general test standards of §160-4 of this Subdivision and all of the following specific test standards.

(1) Test substance. These studies shall be conducted with each active ingredient in the product.

(i) Where radioisotopic analytical techniques are used (they are preferred), studies shall be conducted with the analytical grade of each active ingredient in the product.

(ii) Where non-radioisotopic analytical techniques are used, studies shall be conducted with the technical or purer grade of each active ingredient in the product.

(2) Test procedures. (i) Studies should be conducted at 25± 1°C.

(ii) One or more concentrations of the test substance should be used for this study at levels which will define the kinetics of the reaction and permit isolation and identification of photoproducts formed. For pesticides of low water solubility, an appropriate solubilizing co-solvent may be added to increase water solubility. However, use of such agents should be avoided whenever possible, and, if used, the concentration of co-solvent in the final solution should not exceed 1 percent by volume. The cosolvent should not act as a photosensitizer in the rate study.

(iii) Precautions should be taken during the test to minimize loss of test substance through volatilization.

(iv) Photolysis rate determination and photoproduct identification experiments should be carried out at a pH that minimizes hydrolytic breakdown as determined in § 161-1. The pH selected should be maintained by the use of an appropriate buffer. The buffer solution should be prepared in distilled or deionized water free of all live bacteria, and the glassware should be sterilized to minimize the possibility of microbial degradation of the test substance. For compounds that reversibly ionize or protonate within the pH range of environmental concern, aqueous photolysis

rate determination studies (only) shall be carried out at pH's of 5, 7, and 9.

(v) Samples should be exposed to either natural or simulated (including UV greater than 290 nm wavelength) sunlight conditions. If high intensity radiation studies are conducted to allow shorter testing periods (e.g., use of Crosby reactor, as described in reference (i) of paragraph (e) of this section), data should be provided relating the intensity of the radiation used to that of natural sunlight.

(vi) Non-irradiated samples of one or more concentrations of test substance in water held in darkness should be used as experimental controls.

(vii) Aliquots for analysis should be taken at four or more sampling time intervals, with at least one observation made after one-half of the test substance is degraded or after the equivalent of 30 days natural sunlight (12 hours of light per day), whichever comes first.

(viii) A supplemental rate and photoproduct identification study may also be carried out in the presence of a photosensitizer.

(d) Reporting and evaluation of data. In addition to the applicable information specified in § 160-5, the test report should contain the following specific information:

(1) If sunlight is used as the light source, a record of the intensity of incident sunlight, time of exposure, latitude, time of year, atmospheric cover, and other major variables which affect incident light.

(2) If artificial light is used as the light source, the nature of the source, intensity, wave length distribution, and time of exposure, as well as the relationship of the light intensity employed to that of natural sunlight.

(3) Identification of each photoproduct produced in greater than 10 percent yield at any point during the course of the study, and material balance and half-life estimates for the parent substance.

(e) References. The following references contain experimental procedures for conducting water photolysis studies:

(i) Nakagawa, M., and D.G. Crosby. 1974. Photodecomposition of nitrofen. J. Agr. Food Chem. 22:849-853. [This detailed study provides procedures for photolysis of a pesticide in water under either artificial or natural sunlight.]

(ii) Newsom, H.C., and W.G. Woods. 1973. Photolysis of the herbicide dinitramine (N^3,N^3-diethyl-2,4-dinitro-6-trifluoromethyl-m-phenylenediamine). J. Agr. Food Chem. 21:598-601. [This article contains procedures for study of pesticide photolysis in natural water.]

(iii) Niles, G.P., and M.J. Zabik. 1975. Photochemistry of bioactive compounds. Multiphase photodegradation and mass spectral analysis of basagran. J. Agr. Food Chem. 23:410-415. [Procedures for photolysis in aqueous solution, on soil, and as a thin film are discussed in this article.]

(iv) Su, G.C.C., and M.J. Zabik. 1972. Photochemistry of bioactive compounds. Photolysis of arylamidine derivatives in water. J. Agr. Food Chem. 20:320-323. [Procedures here are well defined and useful for a laboratory study of pesticide photolysis in natural water.]

(v) (Reserved)

(2) Supplemental information for developing a protocol to conduct a water photolysis study for reentry assessment is given in the following references:

(i) Wolfe, N.L., R.G. Zepp, G.L. Baughman, R.C. Fincher, and J.A. Gordon. 1976. Chemical and Photochemical Transformation of Selected Pesticides in Aquatic Systems. Ecological Research Series. USEPA 600/3-76-067. 141 pp. [This publication contains reviews and experiments on the transformation of various pesticides in water. Extensive information is presented that may be useful for the design of experiments and evaluation of data.]

(ii) Under the Toxic Substances Control Act, the Agency has proposed test standards for public review regarding photolysis (and certain other studies). These may be found in 44 FR 16240 (March 16, 1979), entitled "Toxic Substances Control: Discussion of Premanufacturing Testing Policy and Technical Issues; Request for Comment."

§ 161-3 Photodegradation studies on soil.

(a) Purpose. Pesticides are applied to the surface of soil and/or on the exposed surfaces of plants, and are then subject to photodegradation. This study will provide data on photolytic pesticide dissipation and on the nature and persistence of photoproducts formed by soil surface catalyzed photolysis.

(b) When required. Data from a photodegradation study on soil surfaces are required by 40 CFR § 158 to support the registration of an end-use product intended for orchard crop use, field and vegetable crop use, or forestry use. Data from such a study are also required to support each application for registration of a manufacturing-use product which may legally be used to formulate such an end-use product. However, such data are not required to support the registration of end-use products with uses involving application to soils solely by injection of the product into the soil or by incorporation of the product into the soil upon application. See, specifically 40 CFR §158.50 and §158.130 to determine whether these data must be submitted. Section II-A of this Subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(c) Test standards. Soil photodegradation data submitted in response to 40 CFR § 158.130 should be derived from tests which comply with the general test standards of § 160-4 of this Subdivision and all of the following specific test standards:

(1) Test substance. These studies shall be conducted with each active ingredient in the product.

(i) Where radioisotopic analytical techniques are used (they are preferred), studies shall be conducted with the analytical grade of each active ingredient of the product.

(ii) Where non-radioisotopic analytical techniques are used, studies shall be conducted with the technical or purer grade of each active ingredient in the product.

(2) Test procedures. (i) One or more concentrations of the test substance should be used for this study at levels that will permit isolation and identification of photoproducts formed.

(ii) One of the soils (e.g., sandy loam, silt loam, or other soil appropriate to the application site) specified in § 162-1 (Aerobic soil metabolism study) should be used, if data from that study are also submitted.

(iii) Samples of soil should be exposed to either natural or simulated sunlight conditions.

(iv) Samples of soil treated with the pesticide at the same application rate as irradiated soil samples and maintained in darkness should be used as experimental controls.

(v) Soil samples should be taken for analysis at four or more sampling time intervals, with at least one observation made after

one-half of the test substance has degraded or 30 days, whichever comes first. The maximum duration of the study need not exceed 30 days.

(d) Reporting and evaluation of data. In addition to the applicable information specified in § 160-5, the test report should contain the following specific information:

(1) If sunlight is used as the light source, a record of the intensity of incident sunlight, time of exposure, latitude, time of year, atmospheric cover, and other major variables which affect incident light.

(2) If artificial light is used as the light source, the nature of the source, intensity, wave length, distribution, and time of exposure.

(3) Identification of each photoproduct produced in greater than 10 percent yield at any point during the course of the study, and material balance and half-life estimates for the parent substance.

(e) References. (1) The following references contain experimental procedures for conducting soil photolysis studies:

(i) Koshy, K.T., A.R. Friedman, A.L. van der Slik, and D.R. Graber. 1975. Photolysis of benzoic acid 2-(2,4,6-trichlorophenyl)-hydrazide. J. Agr. Food Chem. 23:1084-1088. [A procedure for photolysis of a thin film of pesticide by artificial light is described in this paper.]

(ii) Niles, G.P., and M.J. Zabik. 1975. Photochemistry of bioactive compounds. Multiphase photodegradation and mass spectral analysis of basagran. J. Agr. Food Chem. 23:410-415. [Procedures for photolysis in aqueous solution, on soil, and as a thin film are discussed in this article.]

(2) Under the Toxic Substances Control Act, the Agency has proposed test standards for public review regarding photolysis (and certain other studies). These may be found in 44 FR 16240, (March 16, 1979), entitled "Toxic Substances Control: Discussion of Premanufacturing Testing Policy and Technical Issues; Request for Comment." This discussion can provide supplemental information for developing a protocol to conduct a soil photolysis study.

§ 161-4 Data requirements for photodegradation studies in air.

(a) Purpose. When pesticides are used, humans and other non-target organisms could be exposed to relatively high concentrations of pesticide photoproducts in air. This study will provide data on photolytic pesticide dissipation and on the nature and persistence of photoproducts formed from pesticides in the vapor phase.

(b) When required. Data from a laboratory photodegradation study in the vapor phase are required by 40 CFR § 158 on a case-by-case basis to support the registration of an end-use product intended for orchard or field vegetable crop uses that involve potentially significant exposure to workers. Data from such a study are also required to support each application for registration of a manufacturing-use product which legally could be used to formulate such an end-use product. The Agency will make an assessment of what constitutes a significant inhalation exposure of workers based on the information required by § 163-2(b)(2) of this Subdivision. See, specifically, 40 CFR 158.50 and 158.130 to determine whether these data must be submitted. Section II-A of this Subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(c) Test standards. Air photodegradation data submitted in response to 40 CPR § 158.130 should be derived from tests which comply with the general test standards in § 160-4 and all of the following specific test standards:

(1) Test substance. These studies shall be conducted with one of the following test substances:

(i) Where radioisotopic analytical techniques are used (they are preferred), studies shall be conducted with the analytical grade of each active ingredient in the product; or

(ii) Where non-radioisotopic analytical techniques are used, studies shall be conducted with the technical or purer grade of each active ingredient in the product.

(2) Test procedures. (i) One or more concentrations of the test substance should be used for this study and at levels that will permit isolation and identification of photoproducts formed.

(ii) Air samples should be exposed to a spectrum of light providing or simulating expected use conditions. Temperature should be held relatively constant at 30°C.

(iii) Samples of air treated with the pesticide at the same application rates for irradiated samples and maintained in darkness should be used as experimental controls.

(iv) Air samples should be analyzed at four or more sampling time intervals with at least one observation made after one-half of the test substance has degraded or 30 days, whichever comes first. The maximum duration of the study need not exceed 30 days.

(d) Reporting and evaluation of data. In addition to the applicable information specified in § 160-5, the test report should contain the following specific information:

(1) If sunlight is used as the light source, a record of the intensity of incident sunlight, time of exposure, and other major variables which affect incident light such as latitude, time of year, and atmospheric cover.

(2) If artificial light is used as the light source, the nature of the source, intensity, wave length distribution, and time of exposure.

(3) Identification of each photoproduct produced in greater than 10 percent yield at any point during the course of the study, and material balance amd half-life estimates for the parent substance.

(e) References. The following references contain information for developing a protocol to conduct an air photolysis study:

(1) Crosby, D.G., and K.W. Moilanen. 1974. Vapor-phase photodecomposition of aldrin and dieldrin. Arch. Environ. Contam. Toxicol. 2:62-74. [The reaction vessel described here allows the investigation of vapor phase photolysis while minimizing the effect of reactions on vessel walls.]

(2) Under the Toxic Substances Control Act, the Agency has proposed test standards for public review regarding photolysis (and certain other studies). These may be found in 44 FR 16240, (March 16, 1979), entitled "Toxic Substances Control: Discussion of Premanufacturing Testing Policy and Technical Issues; Request for Comment."

Series 162: METABOLISM STUDIES

§ 162-1 Aerobic soil metabolism studies.

(a) Purpose. Pesticides which come into contact with soil can be metabolically transformed. The purpose of soil metabolism studies is to determine the nature and extent of formation of pesticide degradation products to which rotational crops and nontarget organisms will be exposed, and to facilitate assessment of potential disposal problems.

(b) When required. Data from an aerobic laboratory soil metabolism study are required by 40 CFR § 158 to support the registration of an end-use product intended for terrestrial use or forestry use, and to support each application for registration of a manufacturing-use product which may legally be used to formulate such an end-use product. See, specifically, 40 CFR § 158.50 and § 158.130 to determine whether these data must be submitted. Section II-A of this subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general use.

(c) Test standards. Aerobic Soil metabolism data submitted in response to 40 CFR § 158.130 should be derived from tests which comply with the general tests standards in § 160-4 and all of the following test standards:

(1) Test substance. The study shall be conducted using each active ingredient in the product.

(i) If radioisotopic analytical techniques are used, (they are preferred), the study shall be conducted with the analytical grade of each active ingredient in the product.

(ii) If non-radioisotopic analytical techniques are used, the study shall be conducted with the technical or purer grade of each active ingredient in the product.

(2) Test procedures. (i) Rate, type, and degree of metabolism of the pesticide and its major degradates should be determined in a sandy loam, silt loam, or other soil representative of the soil at the intended application sites.

(ii) One or more concentrations of the test substance should be added to soil at levels sufficient to permit measurement of the disappearance of parent compound and identification of major degradates formed.

(iii) Treated soil should be maintained at any constant temperature between 18 and 30°C with a recommended soil moisture content at 75 percent of 0.33 bar moisture.

(iv) Preferred soil sampling times are at pre-treatment, at 1, 3, 7, and 14 days, and 1, 2, 3, 4, 6, 9 and 12 months post-treatment.

(v) Sufficient soil samples should be taken at each sampling interval to ensure interpretable results.

(vi) Residues occurring at a level of 0.01 ppm or greater at normal field application rates under the label treatment schedule should be identified when feasible. The Agency realizes that searching for and identifying unknown compounds at the 0.01 ppm level in environmental substrates is possible for some but not all compounds. Therefore, the 0.01 ppm level is to be taken as a suggested goal to be met or surpassed. However, registration applicants will not be penalized for not being able to meet the 0.01 ppm goal due to limitations of the analytical method.

(vii) Data should be collected until patterns of decline of the test substance and patterns of formation and decline of degradation products are established in soil, or for one year, whichever comes first, when terrestrial crop, noncrop, and forestry uses are involved. An aerobic soil metabolism study extending to two half-lives of the test substance or to six months duration, whichever comes first, is required for pesticides intended only for greenhouse uses and/or domestic outdoor uses.

(d) Reporting and evaluation of data. In addition to the applicable reporting requirements specified in § 160-5, the test report should contain the following specific information:

(1) Representative residue data for soil samples analyzed in accordance with paragraph (c)(2) of this section;

(2) Soil temperature;

(3) Description of soil source and characteristics;

(4) Material balance;

(5) Half-life estimates;

(6) Identity of residues occurring at levels of 0.01 ppm or greater; and

(7) Residue decline curves.

(e) References. (1) The following references contain experimental procedures for conducting aerobic soil metabolism studies:

(i) Bartha, R., and D. Pramer. 1965. Features of a flask and method for measuring the persistence and biological effects of pesticides in soil. Soil Sci. 100:68-70. [The apparatus described in this paper provides a simple, rapid method for demonstrating microbial degradation, persistence, and relative toxicity of a xenobiotic in soil under laboratory conditions.]

(ii) Betts, P.M., C.W. Giddings, and J.R. Fleeker. 1976. Degradation of 4-aminopyridine in soil. J. Agr. Food Chem. 24:571-574. [This paper contains procedures for study of radio-labeled pesticide in soils and in cultures of soil microorganisms.]

(iii) Ferris, Ian G., and E. Paul Lichtenstein, 1980. Interactions between agricultural chemicals and soil microflora and their effects on the degradation of [14C]-parathion in a cranberry soil. J. Agric. Food Chem. 28:1011-1019. [This paper illustrates the importance of interactions of soil microorganisms and agricultural chemicals in the degradation of other pesticides and contains procedures for necessary investigations The procedures include methods for treatments and incubation of soil, for trapping of volatile products, for extraction of materials from soil, and for analyses.]

(iv) Fleeker, J.R., H.M. Lacy, I.R. Schultz, and E.C. Houkom. 1974. Persistence and metabolism of thiophanate-methyl in soil. J. Agr. Food Chem. 22:592-595. [The soil metabolic procedures in this paper are recommended for development of a protocol.]

(v) Parr, J.F., and S. Smith. 1969. A multipurpose manifold assembly: Use in evaluating microbiological effects of pesticides. Soil Sci.107:271-276. [The procedure here is useful for soil respiration studies, and the style and directions are clear.]

(vi) Parr, J.F. and S. Smith. 1973. Degradation of trifluralin under laboratory conditions and soil anaerobiosis. Soil Sci. 115:55-63. [This paper contains a procedure for the investigation of metabolism of a pesticide in soil using the apparatus discussed in Parr and Smith (1969). The procedures contained in this paper are reported to allow separation of effects of volatilization, photolysis, and nonbiological degradation from microbiological metabolism with respect to dissipation of a pesticide from soil.]

(vii) Schooley, D.A., K.M. Creswell, L.E. Staiger, and G.B. Quistad. 1975. Environmental degradation of the insect growth regulator isopropyl (2E,4E)-11-methoxy-3,7,11-trimethyl2,4-dodecadienoate (methoprene). IV. Soil Metabolism. J. Agr. Food Chem. 23:369-373. [This is an excellent study, and contains procedures for investigation of metabolism of a pesticide in soil.]

(viii) Smith, R.A., W.S. Belles, K.W. Shen, and W.G. Woods. 1973. The degradation of dinitramine (N^3,N^3-diethyl 2,4-dinitro-6-trifluoromethylm-phenylenediamine) in soil. Pest. Biochem. Physiol.

3:278-288. [The procedures reported in this study were well planned and executed, and they describe the required tests.]

(ix) Starr, R.I., and D.J. Cunningham. 1975. Leaching and degradation of 4-aminopyridine-^{14}C in several soil types. Arch. Environ. Contam. Toxicol. 3:72-83. [This paper provides a procedure for a soil metabolism study with a radio-labeled pesticide using soils varying in pH, organic matter content, and sand-silt-clay content.]

(2) Supplemental information for developing a protocol for the determination of non-extractable residues is given in the following references: (i) Kazano, H., P.C. Kearney, and D.D. Kaufman. 1972. Metabolism of methylcarbamate insecticides in soils. J. Agr. Food Chem. 20:975-979. [Among the points of special consideration in this study are the reporting of soil characteristics, the flow sheet and detail of experimental work, and the reporting of recoveries of parent compound and metabolite from soil.]

(ii) Stevenson, F.J. 1965. Gross Chemical Fractionation of Organic Matter. Pp. 1409-1421 in Methods of Soil Analysis. C.A. Black, D.D. Evans, J.L. White, L.E. Ensminger, and F.E. Clark (eds.). Amer. Soc. Agron. Publ. No. 9. Madison, Wisconsin. [The procedures outlined in this publication may be useful for isolation of and accounting for pesticide added to soil. However, care must be exercised when interpreting metabolism from extracts done with acid or alkali, since artifacts can arise with such drastic extraction procedures.]

§ 162-2 Anaerobic soil metabolism studies.

(a) Purpose. The purpose of an anaerobic soil metabolism study is to determine the rate and pattern of pesticide metabolism under anaerobic conditions. This information is used to ascertain effects of flooding or waterlogging on a well-aerated soil, a condition which can have an effect on many oxidation-reduction systems that, in turn, may indirectly affect pesticide metabolism and fate.

(b) When required. (1) Data from an anaerobic soil metabolism study are required by 40 CFR § 158 to support the registration of an end-use product intended for field-vegetable crop use, and to support each application for registration of a manufacturing-use product which any legally be used to make such an end-use product. See, specifically, 40 CFR § 158.50 and §158.130 to determine whether these data must be submitted. Section II-A of this Subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(2) Data from an anaerobic soil metabolism study need not be submitted if data from the anaerobic aquatic metabolism study described in § 162.3 of this subdivision have been submitted.

(c) Test standards. Anaerobic soil metabolism data submitted in response to 40 CFR § 158.130 should be derived from tests which comply with the general test standards in § 160-4 and all of the following specific their test standards:

(1) Test substance. This study shall be conducted using each active ingredient in the product.

(i) If radioisotopic analytical techniques are used (they are preferred), studies shall be conducted with the analytical grade of each active ingredient in the product.

(ii) If non-radioisotopic analytical techniques are used, studies shall be conducted with the technical or purer grade of each active ingredient in the product.

(2) Test procedures. (i) The study should be conducted on the same soil selected for testing in the aerobic soil metabolism study, § 162-1, if data from that study are also submitted.

(ii) A sample of the soil treated with one or more concentrations of the test substance and incubated for 30 days or one half-life (whichever is shorter) should be converted from aerobic to anaerobic conditions by either water-logging or purging the soil with inert gases. Where organic content of the soil is deficient, organic amendments should be supplied to the treated soil within 30 days to ensure a substrate for anaerobic metabolism.

(iii) Treated soil should be maintained at a constant temperature between 18 and 30°C after anaerobic conditions have been established. (The temperature chosen should be the same as that selected in the aerobic soil metabolism study § 162-1, if that study is also required.)

(iv) Preferred sampling intervals are 30 and 60 days after anaerobic conditions have been established.

(v) The length of the study need not extend beyond 60 days.

(vi) Residues occurring at a level of 0.01 ppm or greater at normal field application rates under the label treatment schedule should be identified, when feasible [refer to § 162-1(c)(2)(vi)].

(d) Reporting and evaluation of data. In addition to the applicable reporting requirements specified in § 160-5, the test report should contain the following specific information:

(1) Representative residue data for soil samples analyzed in accordance with paragraph (c)(2) of this section.

(2) Selected soil temperature;

(3) Description of soil source and characteristics;

(4) Material balance;

(5) Half-life estimates;

(6) Identity of residues occurring at levels of 0.01 ppm or greater;

(7) Residue decline curves.

(e) References. (1) The following reference contains experimental procedures for conducting anaerobic soil metabolism studies:

Gowda, T.K.S., and N. Sethunathan. 1976. Persistence of endrin in Indian rice soils under flooded conditions. J. Agr. Food Chem. 24:750-753. [Procedures are reported in this publication for the study of relative persistence of a pesticide in several flooded soils. This paper also provides information of the effects of pH, organic matter content, and microorganism metabolism on persistence of the compound.]

(2) Refer to § 162-1(e) for further references on methods to conduct soil metabolism studies.

§ 162-3 Anaerobic aquatic metabolism studies.

(a) Purpose. The purpose of an anaerobic aquatic metabolism study is to assess the nature and extent of formation of pesticide residues in water and in hydrosoil, since these residues may then be taken up by irrigated crops and passed on to other parts of the aquatic food web. Anaerobic conditions are more likely to prevail where pesticides are found in aquatic environments than in strictly terrestrial environments.

(b) When required. Data from an anaerobic aquatic metabolism study are required by 40 CFR § 158 to support the registration of an end-use product intended for aquatic use, forestry use, or for any aquatic impact uses involving direct discharges of treated

water into outdoor aquatic sites. Data from such a study are also required to support each application for registration of a manufacturing-use product which may legally be used to make such an end-use product. See, specifically, 40 CFR § 158.50 and § 158.130 to determine whether these data must be submitted. The anaerobic soil metabolism study in § 16-22 of this Subdivision may not be substituted for this study. Section II-A of this subdivision contains an additional discussion of the "Formulators' Exemption" and who must submt the required data as a general rule.

(c) Test standards. Anaerobic aquatic-metabolism data submitted in response to 40 CFR § 158.130 should be derived from tests which comply with the general test standards in § 160-4 and all of the following test standards:

(1) Test substance. This study shall be conducted using each active ingredient in the product.

(i) If anaerobic analytical techniques are used (they are preferred), studies shall be conducted with the analytical grade of each active ingredient in the product.

(ii) If non-radioisotopic analytical techniques are used, studies shall be conducted with the technical or purer grade of each active ingredient in the product.

(2) Test procedures. (i) Rate, type, and degree of metabolism of the pesticide should be determined in the laboratory in water plus sediment obtained from and representative of that found at an intended use site. The preferred substrate for this laboratory study is sediment covered with water, but the use of a flooded soil may be adequate.

(ii) If the test is performed using flooded soil, oxygen depletion should be established by flooding for 30 days prior to adding the test substance. The test substance should be applied at a rate sufficient to permit measuring the disappearance of the parent compound and identification of major degradates. Where organic content of the soil is deficient, organic amendments should be supplied within 30 days of application of test substance to ensure a substrate for anaerobic metabolism.

(iii) Treated soil/sediment should be maintained at any constant temperature between 18 and 30°C.

(iv) Data should be collected until patterns of decline of the test substance and patterns of formation and decline of degradation products are established in water and sediment, or for one year, whichever comes first.

(v) Residues occurring at a level of 0.01 ppm or greater at normal field application rates under the label treatment schedule should be identified, when feasible. [Refer to § 161-1(c)(2)(vi)]

(d) Reporting and evaluation of data. In addition to the applicable reporting requirements specified in § 160-5, the test report should contain the following specific information:

(1) Representative residue data for water and sediment samples analyzed in accordance with paragraph (c)(2) of this section.

(2) Soil/sediment temperature, and

(3) Description of soil/sediment/water source and characteristics;

(4) Material balance;

(5) Half-life estimates;

(6) Identity of residues occurring at levels of 0.01 ppm or greater; and

(7) Residue decline curves.

(e) References. The following references contain experimental procedures for conducting anaerobic aquatic metabolism studies:

(i) Gowda, T.K.S., and N. Sethunathan. 1976. Persistence of endrin in Indian rice soils under flooded conditions. J. Agr. Food Chem. 24:750753. [Although the procedure used in this study can provide useful information, it is not necessary to measure the redox potential to establish anaerobiosis if the 30-day flooding requirement is met.]

(ii) Hance, R.J., and G. Chesters. 1969. The fate of hydroxyatrazine in a soil and a lake sediment. Soil Biol. Biochem. 1:309-315. [Although this is a study of a metabolite rather than a pesticide, it is a useful model to illustrate use of a sediment and a soil, establishment of anerobiasis by use of streams of nitrogen or air, comparison of aerobic and anaerobic aquatic metabolism, and use of sterile and inoculated media for study of metabolism with a radio-labeled compound.]

(iii) Miyazaki, S., H.C. Sikka, and R.S. Lynch. 1975. Metabolism of dichlobenil by microorganisms in the aquatic environment. J. Agr. Food Chem. 23:365-368. [This report provides a good model on which to base experimental protocols.]

§ 162-4 Aerobic aquatic metabolism studies.

(a) Purpose. The purpose of aerobic aquatic metabolism studies is to determine the effects on a pesticide of exposure to aerobic conditions in water or sediment during the period of dispersal of the pesticide throughout the aquatic environment, and to compare rates and formation of metabolites with those observed under conditions of anaerobic aquatic metabolism.

(b) When required. Data from an aerobic aquatic metabolism study are required by 40 CFR §158 to support the registration of an end-use product intended for aquatic use or for any aquatic impact uses involving direct discharges of treated water into outdoor aquatic sites, and to support each application for registration of a manufacturing-use product which may legally be used to formulate such an end-use product. See, specifically, 40 CFR § 158.50 and § 158.130 to determine whether these data must be submitted. Section II-A of this Subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(c) Test standards. Aerobic aquatic metabolism data submitted in response to 40 CFR § 158.130 should be derived from tests which comply with the general test standards in § 160-4 and all of the following specific test standards:

(1) Test substance. This study shall be conducted using each active ingredient in the product.

(i) If radioisotopic analytical techniques are used (they are preferred), studies shall be conducted with the analytical grade of each active ingredient in the product.

(ii) If non-radioisotopic analytical techniques are used, studies shall be conducted with the technical or purer grade of each active ingredient in the product.

(2) Test procedures. (i) Pesticide metabolism should be determined in the laboratory in water plus sediment obtained from and representative of that found at an intended use site. The test substance should be added to water at a rate sufficient to permit measuring the disappearance of the parent compound in water and identification of major degradates in water and sediment.

(ii) The treated water/sediment should be maintained at a constant temperature between 18 and 30 °C. (The temperature chosen should be the same as that selected in the anaerobic aquatic metabolism study, § 162-3, if that study is also being conducted.)

(iii) Data should be collected until patterns of decline of the test substance and patterns of formation and decline of degradation products are established in water and sediment or for 30 days, whichever comes first.

(iv) Residues occurring at a level of 0.01 ppm or greater at normal field application rates under the label treatment schedule should be identified, when feasible [Refer to § 162-1(c)(2)(vi)].

(v) For additional procedures and supplementary information on aerobic aquatic metabolism studies, see § 162-4(e).

(d) Reporting and evaluation of data. In addition to data submitted in response to the applicable reporting requirements specified in § 16-05, the test report should contain the following specific information:

(1) The results of analysis of each water and sediment sample required in paragraph (c)(2) of this section;

(2) Soil/sediment temperature;

(3) Description of soil/sediment/water source and characteristics;

(4) Material balance;

(5) Half-life estimates;

(6) Identification of residues occurring at levels of 0.01 ppm or greater; and

(7) Residue decline curves.

(e) References. The following references contain experimental procedures for conducting aerobic aquatic metabolism studies:

(1) Krzeminski, S.F., C.K. Brackett, and J.D. Fisher. 1975. Fate of microbial 3-isothiazolone compounds in the environment: Modes and rates of dissipation. J. Agr. Food Chem. 23:1060-1068.

(2) Krzeminski, S.F., C.K. Brackett, J.D. Fisher, and J.P. Spinnler. 1975. Fate of micccrobicidal 3-isothazolone compounds in the environment: Products of degradation. J. Agr. Food Chem. 23:1068-1075.

[These two papers in combination constitute an excellent model for an aerobic metabolic study. The papers compare degradation and accumulation in several systems and contain an extensive compilation of well-identified metabolites. Of special interest is the inhibition and induction in metabolism reported here.]

(3) Schaefer, C.H., and E.F. Dupras, Jr. 1976. Factors affecting the stability of dimilin in water and the persistence of dimilin in field waters. J. Agr. Food Chem. 24:733-739.

Series 163: MOBILITY STUDIES

§ 163-1 Leaching and adsorption/desorption studies.

(a) Purpose. The movement of pesticide residues by means of leaching through the soil profile or transport to and dispersion in the aquatic environment may cause contamination of food, result in loss of usable land and water resources to man due to contamination of groundwater supplies, or cause habitat loss to wildlife. Therefore, laboratory studies are required to predict:

(1) The leaching potential or pesticides and their degradates through the soil profile at terrestrial sites; and

(2) The movement of pesticides and their degradates to and dispersion in aquatic sites.

(b) When required. Leaching or absorption/desorption data are required by 40 CFR § 158 to support the registration of an end-use product intended for domestic outdoor use, greenhouse use, terrestrial noncrop use, orchard crop use, field-vegetable crop use, forestry use, aquatic use, and aquatic impact uses involving direct discharges of treated water into outdoor aquatic sites. Such data are also required to support each application for registration of a manufacturing-use product which may legally be used to make such an end-use product. See, specifically, 40 CFR § 158.50 and § 158.130 to determine whether these data must be submitted. Section II-A of this Subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(c) Test standards. Leaching or absorption/desorption data submitted in response to 40 CFR § 158.130 should be derived from tests which comply with the general test standards in § 160-4 and all of the following specific test standards:

(1) Test substance. Studies shall be conducted using each active ingredient in the product.

(i) If radioisotopic analytical techniques are used (they are preferred), studies shall be conducted with the analytical grade of each active ingredient in the product;

(ii) If non-radioisotopic analytical techniques are used, studies shall be conducted with the technical or purer grade of each active ingredient in the product.

(2) Test procedure. (i) Analytical technique selection. A laboratory study should be conducted to provide a quantitative estimate of pesticide mobility in soil or absorption/desorption on sediments.

(ii) Amount of test substance. An amount of test substance equal to the highest recommended rate for a single application of the pesticidal active ingredient should be added to the soils/sediment utilized in these studies.

(iii) Soil selection. Each study should include, at a minimum, four soils, such as sand (agricultural), sandy loam, silt loam, clay, or clay-loam, each having a pH within the range of 4-8. However, if the pesticide is to be limited to use with one specific soil type, then the soils selected should include that specific soil type. In addition, if the pesticide is intended for an aquatic use or for an aquatic impact use involving direct discharges of treated water into outdoor aquatic sites, batch equilibrium (adsorption/desorption) studies on one aquatic sediment obtained from or representative of the proposed use area should be provided.

(A) At least one of the soils selected should have an organic matter content less than or equal to one percent (sand or sandy loam preferred).

(B) One of the soils should be the soil used for § 162-1 (aerobic soil metabolism study). This soil preferably should be a sandy-loam soil. This soil shall be used to study leaching of pesticide degradates.

(iv) Preparation of soil for study of pesticide degradates. The test substance should be aged under aerobic conditions for 30 days or one half-life (whichever is shorter) in the soil selected in paragraph (c)(2)(iii)(B) of this section. The treated soil should be maintained at a constant temperature between 18 and 30°C. The temperature chosen shall be the same as that selected in the aerobic soil metabolism study § 162-1) if that study is also required. The treated soil should be maintained at a soil moisture content of 75 percent of 0.33 bar moisture content during the aging period. At the end of the aging period, either a portion of the aged soil containing the pesticide and its degradates or extracts obtained from the aged soil should be tested by one of the methods set forth below:

(A) The extract may be tested on soil thin layer chromatographic (TLC) plates as required by paragraph (c)(2)(v)(A) of this section; or

(B) The portion of aged soil may be added to the prepared soil columns as required by paragraph (c)(2)(v)(B) of this section; or

(C) Alternatively, the mobility of individual degradates which have been demonstratee independently to occur in soil after the aging period specified in paragraph (c)(2)(v) of this section.

(v) Analysis methods. For terrestrial noncrop uses, orchard crop uses, field-vegetable crop uses, and forestry uses, the mobility of the test substance and its degradates in soil should be assessed either by soil thin layer chromatography, soil column, or batch equilibrium (adsorption/desorption) procedures described below in paragraphs (A), (B), and (C), respectively. For domestic outdoor uses, greenhouse uses, aquatic uses, and aquatic impact uses, the mobility of the test substance and its degradates in soil shall be assessed only by the batch equilibrium (adsorption/desorption) procedure. Whatever procedure is selected must be followed for all soils studied.

(A) Soil thin-layer chromatography (TLC) study. Soil TLC studies to predict the leaching potential of pesticides and their degradates in soil should be performed as follows: TLC plates should be prepared using the soils described in paragraph (c)(2)(iii) of this section, to which both the test substance (parent pesticide and degradates) are applied. Application of reference pesticide standards on each TLC plate in addition to the test substance is required, to assess the relative mobility of the test substance to that of other pesticides whose laboratory and field leaching behavior is already known. For experimental procedures on soil and plate preparation, pesticide application, plate development, pesticide visualization, and Rf calculations, see reference (1)(ii) of paragraph (e) of this section.

(B) Soil column study. Soil column studies to define the vertical distribution of the test substance and its degradates in the soil profile should be performed as follows: The column(s) should be from 30 to 300 cm in height, consisting of soils described in § 1631(c)(2)(iii), and should be eluted with a volume of water equal to [groundwater recharge values of] 20 inches (50.8 cm) times the cross sectional area of the column. A distribution curve of the test substance in the column shall be determined by quantification of the test substance and its degradates in 6 cm segments and in the eluate. For experimental procedures on conducting a soil column study, see references (1)(i), (iii), (iv) and (v) of paragraph (e) of this section.

(C) Batch equilibrium (adsorption/desorption) study. Adsorption/desorption coefficients calculated from a batch equilibrium study are used along with solubility data to predict the extent or depth of pesticide leaching in the different soil types tested, and also the extent of pesticide adsorption/desorption on sediments when aquatic or aquatic impact uses are proposed. The study should be conducted using the soils described in paragraph (c)(2)(iii) of this section, plus one representative aquatic sediment (if an

aquatic or aquatic impact use involving direct discharge is proposed). The test substance including its degradates identified in and extracted from the aerobic soil metabolism study (§ 162-1) should be equilibrated with the soils and aquatic sediment selected for this study at four concentrations in a 0.01 N or M Ca ion solution. If necessary, a small amount of acetone or other solvent may be used to achieve solution of poorly soluble pesticides or degradates. For experimental procedures on conducting batch equilibrium (adsorption/desorption) studies, including calculation of Kd values, see references (3)(i) through (xii) and (4)(i) and (ii) of paragraph (e) of this section.

(d) Reporting and evaluation of data. In addition to the applicable reporting requirements specified in § 160-5, the test report should contain the following specific information:

(1) Soil thin layer chromatography (TLC). The mobility of pesticides and their degradates should be reported as mobility class 1 to 5, corresponding to Rf values of 0.0 to 0.09 [immobile (class 1)], 0.10 to 0.34 [low (class 2)], 0.35 to 0.64 [intermediate (class 3)], 0.65 to 0.89 [mobile (class 4)], and 0.90 to 1.0 [very mobile (class 5)], respectively. Values of soil/water relationships (K_d) should be reported using appropriate R_f to K_{oc}/K_d equations. Examples of calculations used in determining K_d values should be provided.

(2) Soil column study. Values of soil/water relationships (K_d) should be reported for the test substance and its degradates using appropriate equations. Examples of calculations used in determining K_d values should be provided.

(3) Batch equilibrium (adsorption/desorption) study. Adsorption/desorption data to be reported should include concentrations of the test substance, including its degradates, partitioned between soil and water and calculated as K_d values from the concentrations using appropriate equations for calculating such values. If the Freundlich equation is used, examples of calculations for 1/n and K values should be provided.

(e) References. (1) The following references contain experimental procedures for conducting mobility (leaching) studies:

(i) Grover, R. 1972. Mobility of dicamba, picloram and 2,4-D in soil columns. Weed Sci. 25:159-162. [This paper compares leaching rates using soil columns and adsorption parameters for several soil-pesticide combinations. It illustrates that theories relating adsorption and movement can be verified with soil columns and the resulting data can be related to field movements.]

(ii) Helling, C.S. 1971. Pesticide mobility in soils. I. Parameters of thin-layer chromatography. Soil Sci. Soc. Amer. Proc. 35: 732-737. [This paper discusses the usefulness of soil thin-layer chromatography and details experimental parameters.]

(iii) Krzeminski, S.F., C.K. Brackett, and J.D. Fisher. 1975. Fate of microbicidal 3-isothiazolone compounds in the environment: modes and rates of dissipation. J. Agr. Food Chem. 23:1060-1068. [This paper contains a procedure for a column leaching study.]

(iv) Lichtenstein, E.P., K.R. Schulz, and T.W. Fuhremann. 1972. Movement and fate of dyfonate in soils under leaching and nonleaching conditions. J. Agr. Food Chem. 20:831-838. [Both GLC and radio-tracer analyses were used in this study with radio-labeling in two positions to allow illustration of different types of degradation products and their movement in soils.]

(v) Weber, J.B., and T.F. Peeper. 1977. Herbicide Mobility in Soils. Pp. 73-78 in Research Methods in Weed Science. B. Truelove (ed.). S. Weed Sci. Soc. Second Edition. Auburn Printing, Inc. Auburn, Alabama. [This paper provides a brief but descriptive analysis of procedures for the study of herbicide leaching in soil, and it discusses the two major types of columns used for these studies.

(2) The following references contain supplemental information pertaining to mobility studies:

(i) Bailey, G.W., and J.L. White. 1970. Factor influencing the adsorption and movement of pesticides in soils. Residue Rev. 32:29-92. [This is a good general review discussing pitfalls to be aware of in planning or interpretation of leaching experiments.]

(ii) Hamaker, J.W., and J.M. Thompson. 1972. Adsorption. Pp. 49-143 in Organic Chemicals in the Soil Environment. Vol. I. C.A.I. Goring and J.W. Hamaker (eds.). Marcel Dekker, Inc., New York. [This is a basic review of the theoretical foundation of pesticide adsorption on soils and an excellent source for equations and the derived constants characterizing pesticidesoil adsorption. The tables of data may aid in initial range-finding.]

(iii) Leistra, M., and W.A. Dekkers. 1976. Computed effects of adsorption kinetics on pesticide movement in soils. J. Soil Sci. 28:340-350. [This is a theoretical paper that may be useful for interpretation of leaching studies, since it illustrates the importance of rainfall pattern with respect to pesticide leaching.]

(iv) Leistra, M., J.J. Smelt, and R. Zanvoort. 1975. Persistence and mobility of bromacil in orchard soils. Weed Res. 15:177-181. [This article is recommended for those planning and interpreting pesticide leaching experiments. It reports that the leaching of a pesticide in field experiments was substantially lower than predicted by calculation from results of soil-TLC experiments.]

(v) Lindstrom, F.T., L. Boersma, and P. Stockard. 1971. A theory on on the mass transport of previously distributed chemicals in a water saturated sorting porous medium: Isothermal cases. Soil Sci. 112:291-300. [This paper presents a theoretical model for predicting pesticide movement from adsorption data and flow rates.]

(vi) Oddson, J.K., J. Letey, and L.V. Weeks. 1970. Predicted distribution of organic chemical in solution and adsorbed as a function of position and time for various chemical and soil properties. Soil Sci. Soc. Amer. 34:412-417. [This paper presents a theoretical model for the movement of pesticides in soils.]

(vii) Van Genuchten, H.T., P.J. Wierenga, and G.A. O'Connor. 1977. Mass transfer studies in sorbing media: III. Experimental evaluation with 2,4,5-T. Soil Sci. Soc. Amer. 41:278-285. [This paper presents comparisons of model calculations and experimental data, and it may be useful for planning or interpretation of leaching experiments.]

(3) The following references contain experimental procedures for conducting adsorption/desorption studies:

(i) Aharonson, N., and U. Kafkafi. 1975. Adsorption, mobility and persistence of thiabendazole and methyl 2-benzimidazolecarbamate in soils. J. Agr. Food Chem. 23:720-724. [The techniques and methods used in this study are useful for both adsorption/desorption and leaching studies.]

(ii) Farmer, W.F., and Y. Aochi. 1974. Picloram sorption by soils. Soil Sci. Soc. Amer. Proc. 38:418-423. [Methods of adsorption and desorption can be found in this study.]

(iii) Grover, R., and R.J. Hance. 1970. Effect of ratio of soil to water on adsorption of linuron and atrazine. Soil Sci. 109:136-138. [This paper presents information on the soil-water ratio to be used in laboratory studies of pesticide adsorption to soils.]

(iv) Hamaker, J.W., C.A.I. Goring, and C.R. Youngson. 1966. Sorption and leaching of 4-amino-3,5,6-trichloropicolinic acid in soils. Advances in Chemistry Series 60:23-37. [The techniques and methods used in this study are standard except for the time allowed for equilibration. The data presented demonstrates the

effect of pH on adsorption of an ionized species and the inverse relationship between partition coefficient and adsorption.]

(v) Hance, R.J. 1967. The speed of attainment of sorption equilibria in some systems involving herbicides. Weed Res. 7:29-36. [This study illustrates the use of range finding experiments to find an equilibration time for meaningful adsorption experiments. Techniques for adsorption and desorption experiments and use of Freundlich isotherms are presented in this paper.]

(vi) Harvey, R.G. 1974. Soil adsorption and volatility of dinitroanilineherbicides. Weed Sci. 22:120-124. [Use of absorption isotherms for the calculation of latent heat of adsorption and the effect of adsorption on volatility of pesticides are illustrated in this paper.]

(vii) Leistra, M., and W.A. Dekkers. 1976. Computed effects of adsorption kinetics on pesticide movement in soils. J. Soil Sci. 28:340-350. [This is a simulation of pesticide behavior on soils in the field by computer.]

(viii) Leistra, M., and W.A. Dekkers. 1977. Some models for the adsorption kinetics of pesticides in soil. J. Environ. Sci. Health B12(2):85-103. [This continuation of the 1976 paper (above) discusses the multimechanism, multirate phenomena responsible for the differences observed in rates of adsorption and rates of desorption.]

(ix) Murray, D.S., P.W. Santelmann, and J.M. Davidson. 1975. Comparative adsorption, desorption, and mobility of dipropetryn and prometryn in soil. J. Agr. Food Chem. 23:578-582. [The correlation between adsorption, cation exchange capacity, organic matter, and clay content is illustrated in this paper, and adsorption/desorption is compared with soil TLC experiments.]

(x) Saltzman, S., L. Kliger, and B. Yaron. 1972. Adsorption/desorption of parathion as affected by soil organic matter. J. Agri. Food Chem. 20:12241226. [The importance of organic matter in absorption of pesticides by soil is discussed in this report.]

(xi) Savage, K.E., and R.D. Wauchope. 1974. Fluometuron adsorption/desorption equilibria in soil. Weeds 22:106-110. [This paper discusses equations used for computation of adsorption isotherms and provides information for equation selection for quantitative depiction of adsorption and desorption.]

(xii) Wu, C.H., N. Buehring, J.M. Davidson, and P.W. Santelmann. 1975. Napropamide adsorption, desorption, and movement in soils. Weed Sci. 23:454457. [Column leaching and soil TLC experiments conducted and reported here are correlated with adsorption/desorption experiments.]

(xiii) (Reserved for: OECD Guidelines for Testing Chemicals. Section 1, Number 106. Adsorption/Desorption.)

(4) The following references contain supplemental information for developing a protocol for adsorption/desorption studies:

(i) Bailey, G.W., and J.L. White. 1970. Factors influencing the adsorption, desorption, and movement of pesticides in soils. Residue Rev. 32: 29-92. [This review provides background information on the principles underlying the processes of adsorption and mobility of pesticides in soil.]

(ii) Weber, J.B. 1977. Soil Properties, Herbicide Sorption, and Model Soil Systems. Pp. 59-72 in Research Methods in Weed Science. 2nd Ed. S. Weed Sci. Soc. B. Truelove (ed). Auburn Printing, Inc. Auburn, Ala. [This is a general review of experimental methods for determination of soil properties, herbicide adsorption, and construction of simple model soil systems.]

§ 163-2 Laboratory volatility studies.

(a) Purpose. Volatilization can be a major mode for the movement of pesticides from treated areas. The vapors resulting from volatilization of some pesticides can cause adverse effects to man via inhalation exposure at sites of application or biological effects in nontarget organisms at some distance from the treated site. The Agency is particularly concerned about commercial greenhouse applications involving intensive use of volatile pesticides, use patterns which are characteristically involved with commodities having high economic value and high labor requirements; such uses can result in significant inhalation exposure to workers and applicators.

(b) When required. (1) Data from a laboratory volatility study are required by 40 CFR § 158 on a case-by-case basis to support the registration of each end-use product intended for commercial greenhouse, orchard, or field-vegetable crop uses that involve significant inhalation exposure to workers. Data from such a study are also required to support each application for registration of a manufacturing-use product which may legally be used to formulate such an end-use product. See, specifically, 40 CFR § 158.50, § 158.130, and the following discussion in § 163.3-2 (b)(2) to determine whether these data must be submitted. Section II-A of this subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(2) The Agency will evaluate the following information provided by the registration applicant to make an assessment of what constitutes a significant inhalation exposure to workers:

(i) Vapor pressure at 25°C and water solubility of the pesticide active ingredient (§§ 63-9 and 63-8 of Subdivision D);

(ii) Soil adsorption coefficient (K_d) of the test substance using the soil from a typical intended application site (§ 163-1 of this Subdivision);

(iii) Soil characteristics, including moisture content, at the intended site of application;

(iv) Method, rate, and intervals of pesticide application;

(v) Temperature, humidity, and air flow rates at the site of application;

(vi) Ventilation sequences or practices for commercial greenhouse applications; and

(vii) Inhalation toxicity of the pesticide (§§ 81-3 and 82-4 of Subdivision F).

(3) The data requirements of this section may also be satisfied by data produced from a study that meets the test standards contained in § 163-3 (field volatility studies).

(c) Test standards. Laboratory volatility data submitted in response to 40 CFR § 158.26 should be derived from tests which comply with the general test standards in § 160-4 and all of the following specific test standards:

(1) Test substance. The test substance shall be a typical end-use product.

(i) If the applicant's product is an end-use product, the test substance shall be a product whose formulation is typical of the formulation category (e.g., wettable powder, emulsifiable concentrate, wettable powder) to which the product belongs.

(ii) If the applicant's product is a manufacturing-use product which legally could be used to make an end-use product for which volatility data are required, the test substance shall be a product representative of the major formulation category which includes that end-use product. (If the manufacturing-use product is usually formulated into end-use products comprising two or more major formulation categories, a separate study must be performed with a typical end-use product for each such category.)

(2) Test procedure. A laboratory study should be conducted to determine the actual rate or extent of pesticide volatilization from soil under controlled conditions only for those pesticides with uses the Agency considers pose a potentially significant

inhalation exposure to workers. Applicants may omit the laboratory studies and perform a greenhouse and/or field study instead. (See § 163-3.)

(i) Laboratory experimental conditions should represent, to the extent possible, an environment where the pesticide is intended for use.

(ii) The rate of test-substance application to soil should approximate the intended rate of field usage.

(iii) The following factors should be addressed in designing a laboratory volatility study:

(A) Properties of the pesticide such as vapor pressure, and water solubility, which can influence the trapping medium and air sampling rates;

(B) Properties relating to the soil, such as adsorption to soil and soil texture, to avoid untoward reduction of the rate of volatility (e.g., sandy soil is preferred);

(C) Environmental factors, such as air temperature, humidity, and movement, to avoid untoward dehydration or flooding of the soil, and to assure efficiency of sampling.

(iv) Air samples should be collected and analyzed for residues in the laboratory experimental equipment used. Monitoring should be conducted continuously or at intervals which increase with time after the start of the experiment. Monitoring should continue until the nature of the residue decline curve has been clearly established.

(d) Reporting and evaluation of data. In addition to the basic reporting requirements specified in § 160-5, the test report should include the following specific information:

(1) Volatility data expressed as $ug/cm^2/hour$;

(2) Air concentrations expressed as ug/m^3 or mg/m^3;

(3) Vapor pressure expressed as torr (or the equivalent expressed in other conventional units);

(4) Temperature and relative humidity;

(5) A description of the soil used; and

(6) A description of the laboratory test equipment used.

(e) References. (1) The following references contain laboratory studies of pesticide volatility; information in these papers could be useful for protocol development:

(i) Kearney, P.C., and A. Kontson. 1976. A simple system to simultaneously measure volatilization and metabolism of pesticides from soils. J. Agr. Food Chem. 24:424-426. [A polyurethane foam trap and a potassium hydroxide trap were used to recover sequentially the parent compound and degradation product from air.]

(ii) Spencer, W.F. and M.M. Claith. 1974. Factors affecting vapor loss of trifluralin from soil. J. Agr. Food Chem. 22:987-991. [The laboratory methods used for determining volatilization of chemicals used in this study allow measurement of effects of several variables. The use of hexane as a trapping medium limits the gas flow rates and volumes that can be used.]

(iii) Spencer, W.F., T.D. Shoup, M.M. Cliath, W.J. Farmer, and R. Haque. 1979. Vapor pressure and relative volatility of ethyl and methyl parathion. J. Agr. Food Chem. 27:273-278. [Polyurethane foam traps and GLC detection largely specific for the compounds of interest were used here. Specific detection avoids interference that may cause falsely high vapor levels in field testing.]

(2) Volatilization studies require methods for the trapping, extraction, cleanup, and quantitation of pesticides. A review of reported methods for laboratory investigations of pesticides in air can be found in:

(i) Lewis, R.G. 1976. Sampling and Analysis of Airborne Pesticides. Pp. 51-94 in Air Pollution from Pesticides and Agricultural Processes. R.E. Lee (ed.). CRC Press, Inc. Cleveland, Ohio.

(ii) [Reserved]

§ 163-3 Field volatility studies.

(a) Purpose. Volatilization can be a major mode for the movement of pesticides from treated areas. The vapors resulting from volatilization of some pesticides can cause adverse effects to man via inhalation exposure at sites of application or biological effects in nontarget organisms at some distance from the treated site. The Agency is particularly concerned about commercial greenhouse applications involving intensive use of volatile pesticides, use patterns which are characteristically involved with commodities having high economic value and high labor requirements; such uses can result in significant inhalation exposure to workers and applicators.

(b) When required. Data from a volatility study conducted on-site in a commercial greenhouse and/or in the field will be required by 40 CFR § 158 on a case-by-case basis only for those pesticides that the Agency considers pose a potentially significant inhalation exposure to workers [see § 163-2(b)] and, based on the results of the laboratory study described in § 163-2, that also demonstrate, in the opinion of the Agency, a significant rate of volatilization from soil. See, specifically, 40 CFR § 158.50 and § 158.130 to determine whether these data must be submitted. Section II-A of this Subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(c) Test standards. Field volatility data submitted in response to 40 CFR § 158.130 should be derived from tests which comply with the general test standards in § 160-4 and all of the following specific test standards:

(1) Test substance. The test substance shall be a typical end-use product.

(i) If the applicant's product is an end-use product, the test substance shall be a product whose formulation is typical of the formulation category (e.g., wettable powder, emulsifiable concentrate, granular product) to which the product belongs.

(ii) If the applicant's product is a manufacturing-use product that legally could be used to make an end-use product for which volatility data are required, the test substance shall be a product representative of the major formulation category which includes that end-use product. (If the end-use products that could be made from the manufacturing-use product belong to two or more major formulation categories, a separate study must be performed for each such category.)

(2) Test procedures. (i) The test substance should be applied to a site which is typical of one of the sites to which the product would be applied.

(ii) The test substance should be applied to soil at the rate and by the method stated in the label directions for the pesticide.

(iii) The following factors should be addressed in designing a greenhouse or field volatility study:

(A) Properties of the pesticide such as vapor pressure and water solubility, which can influence the trapping medium and air sampling rates;

(B) Properties relating to the soil, such as adsorption to soil and soil texture, to avoid untoward reduction of the rate of volatility (e.g., sandy soil is preferred);

(C) Environmental factors, such as air temperature, humidity, and movement, to avoid untoward dehydration or flooding of the soil, and to assure efficiency of sampling.

(iv) Air samples should be monitored for residues at treated sites at intervals which increase with time after pesticide application. For example, the following schedule of sampling times might be appropriate for some situations: 0 and 12 hours, 1, 2, 4, 7, 14, and 21 days. Sampling should be continued until the nature of the dissipation curve has been clearly established.

(d) Reporting and evaluation of data. In addition to information meeting the basic reporting requirements specified in § 160-5, the test report should include the following specific information:

(1) Volatility data expressed as g/ha/day;

(2) Air concentrations expressed as ug/m^3 or ng/m^3;

(3) Vapor pressure expressed as torr (or the equivalent expressed in other conventional units); and

(4) Meterologic conditions (temperature, relative humidity, wind velocity and direction, and cloud cover) during the time of the field study.

(e) References. (1) The following references contain supplemental information for developing a protocol to conduct field volatility studies:

(i) Cliath, M.M., W.F. Spencer, W.J. Farmer, T.D. Shoup, and R. Grover. 1980. Volatilization of S-ethyl N,N-dipropylthiocarbamate from water and wet soil during and after flood irrigation of an alfalfa field. J. Agr. Food Chem. 28:610-613. [This is a well-designed and well-executed field study of volatilization with simultaneous study of other modes of dissipation of a pesticide.]

(ii) Harper, L.A., A.W. White, Jr., R.R. Bruce, A.W. Thomas, and R.A. Leonard. 1976. Soil and microclimate effects on trifluralin volatilization. J. Environ. Qual. 5:236-242. [Ethylene glycol vapor traps and non-specific GLC quantitation were used in this study. The influence of water in soil and thus rainfall during the study on volatilization of a pesticide are illustrated as are effects of wind, turbulence, and temperature.]

(iii) Parmele, L.H., E.R. Lemon, and A.W. Taylor. 1972. Micrometeorological measurement of pesticide vapor flux from bare soil and corn under field conditions. Water, Air, and Soil Pollut. 1:433-451. [This study used hexylene glycol vapor traps and sampling periods adjusted to compensate for decrease in pesticide vapor

concentration during the study. Pesticide vapor flux from soil was calculated and related to micrometeorological measurements.]

(iv) Soderquist, C.J., D.G. Crosby, K.W. Moilanen, J.N. Seiber, and J.E. Woodrow. 1975. Occurrence of trifluralin and its photoproducts in air. J. Agr. Food Chem. 23:304-309. [Although this study was concerned with photolysis of pesticides in air, there are procedures in this paper for measurement of volatilization of a pesticide from soil.]

(2) Volatilization studies require methods for the trapping, extraction, cleanup, and quantitation of pesticides. A review of reported methods for field investigations of pesticides in air can be found in:

(i) Lewis, R.G. 1976. Sampling and Analysis of Airborne Pesticides. Pp. 51-94 in Air Pollution from Pesticides and Agricultural Processes. R.E. Lee (ed.). CRC Press, Inc. Cleveland, Ohio.

(ii) [Reserved]

Series 164: DISSIPATION STUDIES

§ 164-1 Field dissipation studies for terrestrial uses.

(a) Purpose. The purpose of field dissipation studies for pesticides with terrestrial uses is to determine the extent of pesticide residue dissipation under actual use conditions. These studies will generate data required for the evaluation of mobility, degradation, and dissipation of residues. These studies are required because pesticide dissipation may proceed at a different rate under field conditions and therefore result in the formation of levels of degradates differing from those observed in laboratory studies.

(b) When required. Data from a terrestrial field dissipation study are required by 40 CFR § 158 to support the registration of an end-use product intended for any terrestrial use (except greenhouse use), and to support each application for registration of a manufacturing-use product which may legally be used to make such an end-use product. See, specifically, 40 CFR § 158.50 and § 158.130 to determine whether these data must be submitted. Section II-A of this Subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the requiired data as a general rule.

(c) Test standards. Field dissipation data submitted in response to 40 CFR 158.130 should be derived from testing which complies with the general test standards in § 160-4 and all of the following specific test standards:

(1) Test substance. The test substance shall be a typical end-use product.

(i) If an applicant's product is an end-use product, the test substance shall be a product whose formulation is typical of the formulation category (e.g., wettable powder, emulsifiable concentrate, granular product) to which the product belongs.

(ii) If the applicant's product is a manufacturing-use product that legally could be used to make an end-use product for which terrestrial field dissipation data are required, the test substance shall be a product representative of the major formulation category which includes that enduse product. (If the manufacturing-use product is usually formulated into end-use products comprising two or more major formulation categories, a separate study must be performed with a typical end-use product for each such category.)

(2) Test procedures. (i) Sites. Field dissipation studies should be conducted in at least two different sites which are

representative of the areas where the pesticide is expected to be used. For restricted use patterns where only one typical area is involved, data from two similar sites are needed. Studies at additional locations may be needed if the product is intended for a terrestrial crop use, and the sites of application vary appreciably in climate, terrain, or other pertinent characteristics.

(ii) Application. The test substance should be applied using the method of application stated in the directions for use specified on the product label and at the highest rate recommended on the product label.

(iii) Soil sampling. Soil from the treated area should be sampled following treatment for the purpose of ascertaining the extent of pesticide dissipation.

(A) Soil samples serving as test controls should be obtained from the intended application sites immediately prior to application of the test substance and, to the extent possible, from adjacent untreated areas at intervals during the course of the study and at the termination of the study.

(B) Sampling times should include pre-application, date of application, and immediate post-application. In the case of multiple applications, only immediate post-application samples (and not preapplication and date of application samples) should be taken in addition.

(C) Soil samples should be taken in increments, to a maximum depth of 15 cm, provided that the results of studies on pesticide leaching indicate that the test substance is not likely to leach into soil to a depth greater than than 15 cm; and

(D) If data on leaching indicate that the test substance is likely to leach into soil to a depth greater than 15 cm, or if the pesticide is incorporated into soil, then samples should be taken to a depth sufficient to define the extent of leaching.

(iv) Test duration. Residue data should be collected until patterns of decline of the test substance and patterns of formation and decline of degradation products are established in soil, or to the time periods specified below, whichever comes first:

(A) Field and vegetable crop uses: 18 months;

(B) Orchard crop and pastureland uses: 12 months;

(C) Domestic outdoor, park, ornamental, and turf uses: four months; and

(D) Rights-of-way, shelter belts, and related uses: two months.

(d) Reporting and evaluation of data. In addition to the basic reporting requirements specified in § 160-5, the test report should include the following specific information:

(1) Residue decline curves in the tested soil; and

(2) Field test data, including:

(i) Amount of rainfall and irrigation water (accumulated from first application to each sampling);

(ii) Water table;

(iii) Grade (slope);

(iv) Soil and air temperature data;

(v) Techniques and times of planting and harvesting;

(vi) Application time and method;

(vii) Sampling times and techniques;

(viii) Dates and stages of crop and pest development;

(ix) Application-to-harvest (if applicable) and application-to-sampling intervals for each treatment; and

(x) Depth, weight, or volume of each sample taken for analysis.

(e) References. (1) The following references contain information that could be useful for development of a protocol for conducting field dissipation studies:

(i) Caro, J.H., H.P. Freeman, and B.C. Turner. 1974. Persistence in soil and losses in runoff of soil-incorporated carbaryl in a small watershed. J. Agr. Food Chem. 22:860-863. [This is a wellplanned and well-executed field dissipation study.]

(ii) Miller, C.H., T.J. Bnaco, and T.J. Sheets. 1976. Studies on nitralin residues in soils. Weed Sci. 24:288-291. [The experimental design and sampling procedures in this paper are well devised.]

(iii) Polzin, W.J., I.F. Brown, Jr., J.A. Manthey, and G.W. Probst. 1971. Soil persistence of fungicides - Experimental design, sampling, chemical analysis, ad statistical evaluation. Pest. Monit. J. 4:209215. [The factos causing variability in field dissipation studies are considred and analyzed in this paper. However, this study is more detiled than required for pesticide registration.]

(iv) Smith, A.E., and A. Walker. 1977. A quantitative study of asulam persistence in soil. Pestic. Sci. 8:449-456. [The experimental design and statistical analyses of data in this paper are described in detail for field dissipation studies.]

(2) The following reference contains supplemental information for developing a protocol for field dissipation studies:

(i) Goring, C.A.I., D.A. Laskowski, J.W. Hamaker, and R.W. Meikle. 1979. Principles of Pesticide Degradation in Soil. Pp. 135-172 in Environmental Dynamics of Pesticides. R. Haque and V.H. Freed (eds.). Plenum Press. New York. [This is an excellent review for analyses of data and for an understanding of factors affecting persistence of pesticides in soil.]

(ii) [Reserved.]

§ 164-2 Field dissipation studies for aquatic uses and aquatic impact uses.

(a) Purpose. The purpose of aquatic field dissipation studies for pesticides for aquatic uses is to determine the extent of dissipation and mobility of pesticide residues under actual use conditions. These dissipation studies will generate on-site data for evaluating potential hazards of a pesticide under actual use conditions (e.g., mobility, formation of metabolites, and disappearance of parent compound) and provide information with respect to mechanisms of dissipation in various aquatic environments. An aquatic field dissipation study is also required because pesticide dissipation may proceed at a different rate in the aquatic environment than in laboratory aquatic studies.

(b) When required. (1) Except as provided in § 164-2(b)(2), data from an aquatic field dissipation study are required by 40 CFR § 158 to support the registration of an end-use product intended for aquatic food crop uses, for aquatic non-crop uses (which include antifouling paints and other outdoor protective uses where the pesticide-containing surface is in contact with water, and also pesticide application to ditchbanks and shorelines), and for any aquatic impact uses involving direct discharge of treated water into outdoor aquatic sites. Data from such a study are also required to support each application for registration of a manufacturing-use product which may legally be used to make such an end-use product. See, specifically, 40 CFR § 158.50 and § 158.130 to determine whether these data must be submitted. Section II-A of this Subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(2) Pesticides intended for use as antifouling paints and for similar related protective aquatic uses are exempt from the aquatic field dissipation data requirements of 40 CFR § 158.130 if the following conditions are met:

(i) The octanol/water partition coefficient of the product is approximately 1000 or less; and

(ii) The half-life of the active ingredient in water is less than four days as demonstrated by the hydrolysis studies, § 161-1.

(c) Combined testing. Testing conducted to fulfill this data requirement may be combined with the testing conducted to meet the requirements of § 165-4 (field accumulation studies in aquatic nontarget organisms), provided that the test standards for each study are met.

(d) Test standards. Aquatic field dissipation data submitted in response to 40 CFR § 158.130 should be derived from testing which complies with the general test standards in § 160-4, and all of the following test standards:

(1) Test substance. The test substance shall be a typical end-use product.

(i) If the applicant's product is an end-use product, the test substance shall be a product whose formulation is typical of the formulation category (e.g., wettable powder, emulsifiable concentrate, granular product) to which the product belongs.

(ii) If the applicant's product is a manufacturing-use product that legally could be used to make an end-use product for which aquatic field dissipation data are required, the test substance shall be a product representative of the major formulation category which includes that end-use product. [Except for antifouling paints and other related protective-use products (which fall into one formulation category), if the manufacturing-use product is usually formulated into end-use products comprising two or more major formulation categories, a separate study must be performed with a typical end-use product for each such category.]

(2) Test sites. Aquatic field dissipation studies should be conducted in at least two different sites which are representative of the areas where the pesticide is expected to be discharged or applied. For restricted use patterns where only one typical area is involved, data from two similar sites are needed. Studies in additional locations may be needed if the pesticide is intended for an aquatic food crop use, and the sites of application vary in climate, terrain, or other pertinent characteristics.

(3) Application. The test substance should be applied using the method of application stated in the directions for use specified on the product label and at the highest rate recommended on the product label. If the products are for use in pulp and paper mills or industrial cooling towers where direct discharge of pesticide-treated water would be expected, sufficient test substance should be applied to the receiving water in the study to produce the maximum concentration expected for each discharge event.

(4) Sampling. Soil, sediment, and water samples serving as test controls should be obtained from the intended sites of application or from direct aquatic discharges immediately prior to application or discharge of the test substance, and to the extent possible from the adjacent untreated areas, at intervals during the course of the study, and at the termination of the study. Soil, sediment, and water from the treated area should be sampled following treatment for the purpose of ascertaining the extent of pesticide dissipation in accordance with the following:

(i) Sampling times should include pre-application (control), date of application, and immediate post-application for each single or multiple application of the test substance.

(ii) For aquatic food crop uses, soil and water should be sampled.

(iii) For aquatic non-crop uses, soil sediment and water should be sampled.

(iv) For aquatic impact uses resulting in direct discharges, sediment and water should be sampled.

(v) Soil should be sampled in increments to a depth of 15 cm.

(vi) Sediment should be sampled in increments to a depth of 5

(vii) Water should be sampled to a depth dependent upon the use patterns of the pesticide and the site of pesticide action in water (bottom, surface, etc.), and flow meters or comparable techniques shall be used to measure water flow.

(5) Test duration. Residue data should be collected until patterns of decline of the test substance and patterns of formation and decline of degradation products are established in the media samples, or to the maximum time specified below for all use patterns in representative areas, whichever comes first.

(i) Aquatic food crop uses: maximum test duration should be 12 months after application for soil sampling and one month after application for water sampling.

(ii) Aquatic non-crop uses (all use patterns):

(A) Maximum test duration for sediment sampling should be six months for a single application, and for multiple applications, the longer of the following: nine months after the first application, or six months after the last application.

(B) The maximum test duration for water sampling should be one month following each discharge event.

(iii) Aquatic non-crop uses (products intended for application to ditchbanks and shorelines only): the maximum test duration for soil sampling should be six months for a single application, and for multiple applications, the longer of the following: nine months after the first application or six months after the last application.

(iv) Aquatic impact uses resulting in direct discharges:

(A) The maximum test duration for sediment sampling should be six months following a single discharge event, and following multiple discharge events, the longer of the following: nine months after the first discharge or six months after the last discharge.

(B) The maximum test duration for water sampling should be one month following each discharge event.

(e) Reporting and evaluation of data. In addition to the basic reporting requirements specified in § 160-5, the test report should include the following specific information:

(1) Decline curves of residues in each major substrate analyzed; and

(2) Field test data, including:

(i) Dates of planting and harvesting of crops, if applicable;

(ii) Application time(s) and method;

(iii) Sampling times and techniques;

(iv) Dates and stages of crop and pest development, if applicable;

(v) Application-to-harvest (if applicable) and application-to-sampling intervals for each treatment;

(vi) Depth, weight, or volume of each sample, and weights and volumes of aliquots taken for analysis; and

(vii) Flow data expressed in terms of volume or linear flow.

(f) References. (1) The following references contain supplemental information for developing a protocol for conducting aquatic crop studies:

(i) Demint, R.J., J.C. Pringle, Jr., A. Hattrup, V.F. Bruns, and P.A. Frank. 1975. Residues in crops irrigated with water containing trichloroacetic acid. J. Agr. Food Chem. 23:81-84. [This paper presents a procedure for assessment of pesticide carryover in irrigation water.]

(ii) Rice, C.P., H.C. Sikka, and R.S. Lynch. 1974. Persistence of dichlobenil in a farm pond. J. Agr. Food Chem. 22:533-534. [This paper presents procedures for assessment of fate of a pesticide in a water-sediment system.]

(iii) Schaefer, C.H., and E.F. Dupras, Jr. 1976. Factors affecting the stability of dimilin in water and the persistence of dimilin in field waters. J. Agr. Food Chem. 24:733-739. [This paper contains a small-scale technique for assessment of the fate of a pesticide in a water-sediment ecosystem.]

(2) The following references contain supplemental information for developing a protocol for conducting aquatic non-crop studies:

(i) Rice, C.P., H.C. Sikka, and R.S. Lynch. 1974. Persistence of dichlobenil in a farm pond. J. Agr. Food Chem. 22:533-534. [This paper contains a procedure for assessment of fate of a pesticide in a watersediment system. Information on background interference and pesticide recovery from soil and water are presented.]

(ii) Schaefer, C.H., and E.F. Dupras, Jr. 1976. Factors affecting the stability of dimilin in water and the persistence of dimilin in field waters. J. Agr. Food Chem. 24:733-739. [This paper contains a small-scale technique for assessment of fate of a pesticide.]

§ 164-3 Dissipation studies for forestry uses.

(a) Purpose. Data from a residue dissipation study, conducted under actual use conditions, will indicate the extent and rate of pesticide residue dissipation and mobility in both aquatic and terrestrial environments which are encompassed in forestry sites.

(b) When required. Data from a field dissipation study for forestry uses are required by 40 CFR § 158 to support the registration of an end-use product intended for forestry use, and by each applicant for registration of a manufacturing-use product which may legally be used to make such an end-use product. See, specifically,

40 CFR § 158.50 and § 158.130 to determine whether these data must be submitted. Section II-A of this subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(c) Combined testing. Testing conducted to fill this data requirement may be combined with testing conducted in accordance with § 165-5 (field accumulation studies in aquatic nontarget organisms), provided that the test standards for each study are met.

(d) Test standards. Forestry dissipation data submitted in response to 40 CFR § 158.130 should be derived from testing which complies with the general test standards in § 160-4 and all of the following specific test standards:

(1) Test substance. The test substance shall be a typical end-use product.

(i) If the applicant's product is an end-use product, the test substance shall be a product whose formulation is typical of the formulation category (e.g., wettable powder, emulsifiable concentrate, granular product) to which the product belongs.

(ii) If the applicant's product is a manufacturing-use product that legally could be used to make an end-use product intended for forestry use, the test substance shall be a product representative of the major formulation category which includes that end-use product. [If the manufacturing-use product is usually formulated into end-use products comprising two or more major formulation categories, a separate study must be performed with a typical end-use product for each such category.]

(2) Test procedures. (i) Sites. A dissipation study should be conducted in at least one location representative of the areas in which the pesticide product would usually be used. Studies at additional sites may be necessary if the product is intended for use in forest sites with substantially differing characteristics.

(ii) Application. The test substance should be applied using the method of application stated in the directions for use specified on the product label, and at the highest rate recommended on the label.

(iii) Environmental components. The level of pesticide residues shall be measured in the following items:

(A) Foliage (if the pesticide is foliar-applied);

(B) Leaf litter;

(C) Soil under leaf litter;

(D) Exposed soil;

(E) Standing (pond) water;

(F) Moving (stream) water; and

(G) Sediments from both ponds and streams.

(iv) Controls. Test control samples of the environmental components described in paragraph (d)(2)(iii) of this section should be obtained from the intended sites of application immediately prior to application of the test substance and, to the extent possible, from adjacent untreated areas at intervals during the course of the study and at the termination of the study.

(v) Sampling intervals. (A) For exposed soil, soil under leaf litter, and foliage, sampling times should include, at a minimum, preapplication (control), date of application, and three samplings within the first week post application for each single or multiple application of the test substance.

(B) For sediments, standing (pond) water, and moving (stream) water, sampling should include, at a minimum, pre-application (control), date of application, and immediately post-application for each single or multiple application of the test substance.

(v) Test duration. Residue data should be collected until patterns of decline of the test substance and patterns of formation and decline of degradation products are established in the media sampled or the maximum times specified below, whichever comes first: 12 months after the last application for soil and leaf litter, 6 months after the last application for sediment, and 1 month after the last application for water (pond and stream).

(vi) Dissipation curves. Decline curves shall be constructed for residues in leaf litter, soil foliage, and standing water.

(e) Reporting and evaluation of data. In addition to the basic reporting requirements specified in § 160-5, the test report should include the following specific information:

(1) Decline curves of residues in each major substrate analyzed; and

(2) Field test data, including:

(i) Precipitation (accumulated from first application to each sampling);

(ii) Water table;

(iii) Grade (slope);

(iv) Application time;

(v) Sampling time;

(vi) Dates and stages of pest development;

(vii) Application-to-sampling intervals for each treatment;

(viii) Depth, weight, or volume of each sample, and weights and volumes of aliquots taken for analysis; and

(ix) When water flow is measured, flow data expressed in terms of volume or linear flow.

(f) References. The following references contain supplemental information for developing a protocol for conducting forest field dissipation studies:

(1) Symons, P.E.K. 1977. Dispersal and toxicology of the insecticide fenitrothion; predicting hazards of forest spraying. Residue Rev. 68:1-31. [This review provides general information and an overview of problems that have occurred in a forest environment in association with use of an insecticide for control of a forest pest. Pesticide residues in organisms and physical environment and dissipation of the residues are discussed.]

(2) Roberts, J.R., Greenhalgh, R., and Marshall, W.K., eds. 1977. Proceedings of a Symposium on Fenitrothion: The Long-term Effects of its Use in Forest Ecosystems. (Natl. Res. Council Can.: Ottawa, Canada) NRCC/CNRC No. 16073:573-614. [Individual papers within these proceedings contain protocols for the study of pesticide dissipation in a forest ecosystem.]

(3) Giles, R.H., Jr. 1970. The Ecology of a Small Forested Watershed Treated with the Insecticide Malathion-S 35. Wildlife Monographs. No. 24. The Wildlife Society. Washington, D.C. [This paper contains experimental procedures for conducting terrestrial/aquatic (forest) dissipation studies. Studies contained in the reference are more extensive than generally required.]

(4) Maguire, R.J. and E.J. Hale. 1980. Fenitrothion sprayed on a pond: kinetics of its distribution and transformation in water and sediment. J. Agr. Food Chem. 28:372-378. [This paper contains protocols for the study of dissipation of a pesticide from a pond within a forest and from segments within that environment.]

(5) Peiper, G.R. 1979. Residue analysis of carbaryl on forest foliage and in stream water using HPLC. Bull. Environ. Contam. Toxicol. 22:167-171. [This paper contains studies of residue concentrations and dissipation of those residues from plant surfaces and from forest streams after serial pesticide application.]

(6) Szeto, S.Y., H.R. MacCarthy, P.C. Oloffs and R.F. Shepherd, 1978. Residues in Douglas-fir needles and forest litter following an aerial application of acephate (Orthene). J. Environ. Sci. Health (B) 13:87-103. [This paper contains protocols for the study of distribution, transformation, and dissipation of a pesticide within a forest canopy after aerial application.]

§ 164-4 Dissipation studies for combination products and tank mix uses.

(a) Purpose. The objective of this study is to determine the dissipation characteristics of a pesticide in soil when applied as a tank mix or in combination with other pesticides, whether in a combination product or pursuant to labeling directions recommending the simultaneous or serial application of two or more products. The Agency requires these data to ascertain if overall soil persistence of pesticides is affected by the simultaneous or serial application of two or more pesticides.

(b) When required. A laboratory or field soil dissipation study is required by 40 CFR § 158 on a case-by-case basis to support the registration of an end-use product containing more than one active ingredient intended for use as a component in tank mixtures, or customarily applied serially with another pesticide product. See, specifically, 40 CFR § 158.50 and § 158.130 to determine whether these data must be submitted. Section II-A of this subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(c) Test standards. The dissipation studies described in paragraph of this section should be derived from testing which complies with the general test standards in § 160-4 and all of the following specific test standards:

(1) Test substance. (i) For studies on combination products, if the end-use product contains two or more active ingredients (combination product), the test substances shall be the end-use product and similarly formulated products containing each active ingredient singly.

(ii) For studies on tank mixtures, if the use directions state that two or more end-use products containing different active

ingredients may be applied as a tank mixture, the test substances shall be the mixture of the end-use products prepared in accordance with the label directions, and each separate end-use product.

(iii) For studies on serial applications, if the use directions state that two or more end-use products containing different active ingredients may be applied serially, the test substances shall consist of the two or more end-use products to be used in the sequence specified in the label directions.

(2) Test procedures. (i) Application. The test substance(s) should be applied at the highest rate recommended by product labeling and as follows:

(A) For combination products, apply the test substances at the label-recommended field application rates to both light- and heavy-textured soils. Incorporate the test substances into the soil if recommended by label directions.

(B) For tank-mixed pesticides, apply the test substances at the label-recommended field application rates to both light- and heavy-textured soils. Incorporate each test substance into the soil if recommended by label directions.

(C) For serially-applied pesticides, apply the test substances individually and as the sequential combination at the label-recommended field application rates to both light- and heavy-textured soils. Incorporate each test substance into the soil if recommended by label directions.

(ii) Soil sampling. Soil from the treated area or laboratory container should be sampled following treatment for the purpose of ascertaining the extent of pesticide dissipation.

(A) Soil samples obtained from the intended site(s) of application or from the laboratory containers immediately prior to application of the test substance should be used as experimental controls.

(B) Sampling times should include pre-application, date of application, and immediate post-application. In the case of multiple applications, only immediate post-application samples (and not preapplication and date of application samples) are to be taken in addition.

(C) Soil samples should be taken in increments to a depth of 15 cm, unless results of studies on pesticide mobility indicate that the test substance is likely to leach into soil deeper than 15 cm.

(D) If data on leaching indicate that the test substance is likely to leach into soil deeper than 15 cm, or if the pesticide is incorporated into soil deeper than 5 cm, samples should be taken to a depth sufficient to include most of the leached pesticide.

(iii) Test duration. Residue data should be collected until patterns of decline of the test substance and patterns of formation and decline of degradation products are established in soil, or for a maximum duration of six months, whichever occurs sooner.

(d) Reporting and evaluation of data. In addition to the basic reporting requirements specified in § 160-5, the applicable reporting requirements as specified in § 164-1(d) apply.

(e) References. The following references contain supplemental information for developing a protocol to conduct a combination or tank mix study:

(i) Kaufman, D.D., J. Blake, and D.E. Miller. 1971. Methylcarbamates affect acylanilide herbicide residues in soil. J. Agr. Food Chem. 19:204-206. [This paper provides techniques to assess a complex pesticide degradation problem.]

(ii) Kaufman, D.D., P.C. Kearney, D.W. Von Endt, and D.E. Miller. 1970. Methylcarbamate inhibition of phenylcarbamate metabolism in soil. J. Agr. Food Chem. 18:513-519. [This precursor of the 1971 paper (above) contains procedures that may be useful for the design of experimental studies required on combination and tank mixes.]

§ 164-5 Long-term soil dissipation studies.

(a) Purpose. The objective of this study is to enable the Agency to assess the fate of pesticide residues that do not readily dissipate in the soil environment. Slow pesticide dissipation in soil may increase the residue burden imposed by pesticides by increasing their residence time in the environment.

(b) When required. (1) Data from a long-term soil dissipation study are required by 40 CFR § 158 to support the registration of any end-use product:

(i) If it contains an active ingredient with residues that do not reach 50 percent dissipation in soil prior to recommended subsequent application of that same active ingredient to the same sites utilized for the field dissipation study described for field and vegetable crops in § 164-1 and for aquatic food crops in § 164-2(b)(1); or

(ii) If the aerobic soil metabolism study described in § 162-1 demonstrates that, for field and vegetable crop uses and aquatic food crop uses, the total of pesticide (excluding bound) residues in soil are greater than fifty percent of the amount of pesticide initially applied at the time when a subsequent application would occur.

(2) Data from a long-term soil dissipation study are also required to support each application for registration of a manufacturing-use product that legally could be used to make an end-use product meeting the criteria of paragraphs (b)(1)(i) or (ii) of this section. See, specifically, 40 CFR § 158.50 and § 158.130 to determine whether these data must be submitted. Section II-A of this Subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(c) Test standards. Long-term soil dissipation data submitted in response to 40 CFR § 158.130 should be derived from testing which complies with the general test standards in § 160-4 and all of the following test standards:

(1) Test substance. The test substance shall be a typical enduse product.

(i) If the applicant's product is an end-use product, the test substance shall be a product with a formulation typical of the formulation category (e.g., wettable powder, emulsifiable concentrate, granular product) to which the product belongs.

(ii) If the applicant's product is a manufacturing-use product that legally could be used to make an end-use product for which longterm soil dissipation data are required, the test substance shall be a product representative of the major formulation category which includes that end-use product. [If the manufacturing-use product is usually formulated into end-use products comprising two or more major formulation categories, a separate study must be performed with a typical end-use product for each such category.]

(2) Test procedures. The applicant should follow the test procedure applicable to the use pattern or patterns of the product:

(i) Field and vegetable crop uses. (A) The test substance should be applied using the method and highest rate of application stated in the directions for use specified in the product label.

(B) Soil sampling (methods and scheduling) should be performed in accordance with the standards set forth in § 164-1(c)(2)(iii).

(C) Total duration of this study need not exceed three years or extend two years beyond the length of the terrestrial field dissipation study for field and vegetable crop uses.

(ii) Aquatic food crop uses. (A) The test substance should be applied using the method and highest rate of application stated in the directions for use specified on the product label.

(B) Soil sampling (method and scheduling) shall be performed in accordance with the standards set forth in § 164-2(d)(4).

(C) Total duration of this study need not exceed three years or extend two years beyond the length of the aquatic field dissipation study for aquatic food crop uses.

(d) Reporting and evaluation of data. In addition to the applicable reporting requirements specified in § 160-5, the specific reporting requirements described in § 164-1(d) apply for studies involving field and vegetable crop uses, and those in 164-2(e) apply for studies involving aquatic food crop uses.

(e) References. The following references contain supplemental information for developing a protocol to conduct a long-term soil dissipation study:

(1) Burnside, O.C. 1974. Trifluralin dissipation in soil following repeated annual applications. Weed Sci. 22:374-377. [This paper reports a long-term pesticide dissipation study with repeated applications at different levels of treatment and with replication. Assessment of residue levels was accomplished by bioassays with several crops and by a chemical analysis of soil extracts. The use of this bioassay technique can indicate the quantity of active herbicide remaining, but the technique is limited to herbicides. A chemical pesticide residue analysis is more generally applicable.]

(2) Caro, J.H., H.P. Freeman, and B.C. Turner. 1974. Persistence in soil and losses in runoff of soil-incorporated carbaryl in a small watershed. J. Agr. Food Chem. 22:860-863. [This paper is recommended as a model for the design of a long-term study protocol. The use of a field standard applied with the subject pesticide might aid in interpretation of dissipation kinetics.]

(3) Demint, R.J., J.C. Pringle, Jr., A. Hattrup, V.F. Burns, and P.A. Frank. 1975. Residues in crops irrigated with water containing trichloroacetic acid. J. Agr. Food Chem. 23:81-84. [The methods described here may be useful in designing protocols for long-term study of pesticides applied in irrigation water.]

(4) Miller, C.H., T.J. Monaco, and T.J. Sheets. 1976. Studies on nitralin residues in soils. Weed Sci. 24:286-291. [This study evaluated the effect of residue build-up from repeated application of three levels of nitralin to soils. Both biological and chemical assays were used with a sensitive crop for the bioassay. Combination of the two assays may in some cases aid

in interpretation of data. The use of plastic for storing samples should be avoided to prevent possible interferences from desorbed plastic components or loss of residues by sorption to the plastic.]

(5) Polzin, W.J., I.F. Brown, Jr., J.A. Manthey, and G.W. Probst. 1971. Soil persistence of fungicides - Experimental design, sampling, chemical analysis and statistical evaluation. Pest. Monit.J. 4:209215. [This paper was not intended as a long-term study, but rather was a study to improve the reliability of soil persistence data. It is cited here as a guide for improving soil sampling protocols.]

(6) Rice, C.P., H.C. Sikka, and R.S. Lynch. 1974. Persistence of dichlobenil in a farm pond. J. Agr. Food Chem. 22:533-534. [This paper reports dissipation of pesticide from water and the associated sediment in a pond. The use of blanks and fortified samples to evaluate recovery of pesticide from substrates and interference to quantitation as was done in this paper are good practice.]

(7) Schaefer, C.H., and E.F. Dupras, Jr. 1976. Factors affecting the stability of dimilin in water and the persistence of dimilin in field waters. J. Agr. Food Chem. 24:733-739. [Several of the phenomena observed in this paper are worth considering. Dissolution of pesticide formulation can be the rate-limiting step in a pesticide dissipation, and the whole system must be sampled, including strata, to give an accurate material balance. The evaluation of pesticide stability in samples is recommended especially if there will be delay between sampling and analysis.]

Series 165: ACCUMULATION STUDIES

§ 165-1 Confined accumulation studies on rotational crops.

(a) Purpose. Data from confined (laboratory/greenhouse/outdoor small plot) accumulation studies on rotational crops will enable the Agency to determine the nature and amount of pesticide residue uptake in rotational crops. Such data are used to establish realistic crop rotation restrictions (time from application to a time when crops can be rotated) or to provide information for determining if tolerances are needed in rotational crops.

(b) When required. Data from a confined accumulation study on rotational crops are required by 40 CFR § 158 to support the registration of an end-use product intended for field-vegetable crop use, aquatic crop use, or use on any other site on which it is reasonably foreseeable that any food or feed crop may be produced after application of a pesticide. Such data are also required to support each application for registration of a manufacturing-use product which legally could be used to make such an end-use product. See, specifically, 40 CFR § 158.50 and § 158.130 to determine whether these data must be submitted. Section II-A of this Subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(c) Test standards. Rotational crop accumulation data submitted in response to 40 CFR § 158.130 should be derived from testing which complies with the general test standards in § 160-4 and all of the following specific test standards:

(1) Test substance. This study shall be conducted using the radioactively-labeled analytical grade of the active ingredient.

(2) Test procedures. (i) This study should be performed using a sandy loam soil which has been treated with the test substance applied at a rate equivalent to that expected under actual field use conditions. However, if the label instructions of the product limit its use to one soil type other than the sandy loam, then the study should be run with the soil type specified on the label.

(ii) Following soil treatment, the pesticide should be aged under aerobic conditions in the soil for a time approximating the anticipated agricultural practice (e.g., 1 year for crops rotated the following year, 120 days for crops rotated immediately after harvest, and 30 days for assessing circumstances of crop failure). Growing the treated crop in the soil during the aging period is not precluded.

(iii) Crops planted in the treated and aged soil should include those expected in the proposed rotational schedule and, where possible, be representative of each of the following crop groupings: root (e.g., beets, carrots); small grain (e.g., wheat, barley), and leafy vegetable (e.g., spinach, lettuce). The selected crops shall be analyzed for residues at appropriate harvest intervals. (Residue analyses should be performed on selected crops at multiple intervals if both immature and mature crops are normally harvested in the course of usual agricultural practices.)

(iv) Residues in the soil should be analyzed at the time of treatment, at the time of planting of the rotational crop, and at the time of harvest of the rotational crop. However, if this confined accumulation study is carried in the same soil and at the same time as the aerobic soil metabolism study (§162-1) or the terrestrial field dissipation study (§164-1), then the soil residue analyses from these studies will suffice. See also §160-4(e) of this Subdivision.

(d) Reporting and evaluation of data. In addition to the applicable reporting requirements specified in § 160-5, the following data should be reported:

(1) The registration applicant should characterize and, when feasible, identify and provide analytical values for significant residues in the crops tested. Significant residues include parent compound, closely-related degradates, metabolites and/or their conjugates in the crop, but do not include C^{14} activity in the crop associated with that being incorporated into the carbon pool and ultimately into natural plant constituents. In cases where identification of residues is not feasible due to insufficient sample, then pooling of samples obtained from replicate experiments conducted simultaneously should be carried out to enable residue identification to be achieved. From the results of this study, the Agency will determine whether additional studies to measure the accumulation of pesticide residues in rotated crops under actual field conditions are needed. If such field studies are needed, the registration applicant will need to determine whether to conduct the field study described in § 165-2 of this Subdivision or to carry out residue uptake studies described in Subdivision O (Residue Chemistry Data Requirements) necessary to support the establishment of a tolerance in the rotated crop. (See also FR 46(8), 3016, January 13, 1981.)

(2) Depending on the crop tested, separate analyses should be conducted on different portions of the plant. For example, both the aerial and root portions of root crops should be analyzed.

(3) Analyses, including a description of data variability, for residues of parent compound and degradates in soil for each sampling interval.

(4) A description of the growing conditions should be reported. If the study is conducted outdoors, rainfall data, temperature monitoring data, and general climatic conditions should be reported for the test period.

(e) References. The following references contain information for developing a protocol for study of the uptake of residues in rotational crops:

(1) Burnside, O.C. 1974. Trifluralin dissipation in soil following repeated annual applications. Weed Sci. 22:374-377. [The general methods used by this author could be adapted to small-scale field studies. The techniques used in this study should be considered for development of a protocol to obtain data on pesticide uptake with rotational crops.]

(2) Burnside, O.C., C.R. Fenster, and G.A. Wicks. 1971. Soil persistence of repeated annual applications of atrazine. Weed Sci. 19: 290-293. [This paper contains procedures for investigation of pesticide fate with different soils and cropping practices.]

(3) Sirons, G.J., R. Frank, and T. Sawyer. 1973. Residues of atrazine, cyanazine, and their phytotoxic metabolites in a clay loam soil. J. Agr. Food Chem. 21:1016-1020. [This is not a study of uptake of a pesticide in a rotational crop system, but the procedures used could be applicable to design such a study. The study of a phytotoxic metabolite is included in this study.]

(4) Bull, D.L., and G.W. Ivie, 1982. Fate of O-[4-[(4-chlorophenyl)thio]phenyl] O-ethyl S-propyl phosphorothioate (RH-0994) in soil. J. AGR. Food Chem 30: 150-155. [This paper is a good example of procedures for a combined soil metabolism and rotational crop uptake study. The paper differs from the requirements of § 165-1 in that the 14C residues in the rotated crops were not characterized.]

§ 165-2 Field accumulation studies on rotational crops.

(a) Purpose. Data from field accumulation studies on rotational crops will enable the Agency to determine under actual field-use conditions the nature and amount of pesticide residue uptake in rotational crops. Such data are used to establish realistic crop rotation restrictions (time from application to a time when crops can be rotated) and to provide information for determining if tolerances are needed in rotational crops.

(b) When required. Data from a field accumulation study to determine the uptake of soil residues by rotational crops are

required by 40 CFR § 158 to support the registration of an end-use product, and to support each application for for registration of a manufacturing-use product used to make such an end-use product, only under the following circumstances:

(1) When significant C^{14} pesticide residues of concern to the Agency are detected in the test crops analyzed in the confined accumulation study, § 165-1[See § 165-1(d)(1) for a discussion of significant residues]; or

(2) When a subsequent crop is treated with the same active ingredient as the initial crop.

(3) See, specifically, 40 CFR § 158.50 and § 158.130 to determine whether these data must be submitted. Section II-A of this Subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(c) Test standards. Field accumulation data submitted in response to 40 CFR § 158.130 should be derived from testing which complies with the general test standards in § 160-4 and all of the following test standards:

(1) Test substance. The test substance shall be a typical end-use product.

(i) If the applicant's product is an end-use product, the test substance shall be a product whose formulation is typical of the formulation category (e.g., wettable powder, emulsifiable concentrate, granular product) to which the product belongs.

(ii) If the applicant's product is a manufacturing-use product that legally could be used to make an end-use product for which rotational crop accumulation data are required, the test substance shall be a product representative of the major formulation category which includes that end-use product. (If the manufacturing-use product is usually formulated into end-use products comprising two or more major formulation categories, a separate study must be performed with a typical end-use product for each such category.)

(2) Test procedure. (i) Sites. Field accumulation studies should be conducted in at least two different sites which are representative of the areas where rotated crops are expected to be grown. The soil type at one of the test sites should be the same as that used in the confined accumulation study of § 165-1. For restricted use patterns where only one typical area is involved, data from two similar sites should be submitted. See, specifically, 40 CFR § 158.50 and § 158.130 to determine whether these data must be submitted. Section II-A of this Subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(ii) Application. (A) The soil at the test site should be treated with the test substance applied by the method stated in the directions for use specified on the product label and at the highest recommended label rate.

(B) Following soil treatment, the pesticide should be aged under aerobic conditions in the soil for a time approximating the anticipated agricultural practice (e.g., one year for crops rotated the following year, 120 days for crops rotated immediately after harvest, and 30 days for assessing circumstances of crop failure). Growing the treated crop in the soil during the aging period is not precluded.

(iii) Sampling. (A) Representative root, small grain, and leafy vegetable crops typical of the area where the product is to be applied should be planted as rotational crops.

(B) If the registration applicant is proposing a tolerance for residues in a rotated crop, then that crop should be planted, harvested, and analyzed for residues at test sites selected in accordance with the requirements of 40 CFR § 158.125, described in detail in Subdivision O (Residue Chemistry Data Requirements).

(C) The rotational crops should be analyzed for residues at appropriate harvest times. (Residue analyses should be performed on selected crops at multiple intervals if both immature and mature crops are normally harvested in the course of usual agricultural practices.)

(D) Residues in the soil should be analyzed at times of treatment, at time of planting rotational crops, and at the time of rotational crop harvest. These soil analyses may not be needed if the aerobic soil metabolism study (§ 162-1) or the terrestrial field dissipation study (§ 164-1) provide soil residue data that demonstrate essentially complete dissipation of the pesticide by the time the original crop is harvested. (In some cases, this study can be combined with the aerobic soil metabolism study and the terrestrial field dissipation study.)

(iv) Test duration. Residue data should be collected in soil and rotational crops until the time that the rotational crop is normally harvested.

(d) Reporting and evaluation of data. In addition to the applicable reporting requirements specified in § 160-5, the following data should be reported:

(1) Field test data including:

(i) Dates of planting and harvesting;

(ii) Amount of rainfall and irrigation water (accumulated from application to harvest);

(iii) Depth of water table;

(iv) Slope of test site(s);

(v) Temperature monitoring data and a description of the general climatic conditions at the test site during the study;

(vi) Techniques and times of planting, culture, and harvesting;

(vii) Application time and method;

(viii) Sampling times and techniques;

(ix) Stages of crop development at times of sampling;

(x) Application-to-harvest interval; and

(xi) Depth, weight, or volume of each sample taken for analysis.

(2) Analysis for residues of parent compound and degradates in the crops. Depending on the crop, separate analyses should be conducted on different portions of the plant. For example, analysis of both the aerial and root portions of root crops should be conducted.

(3) Analyses for residues of parent compound and degradates in soil for each sampling interval.

(4) A description of residue data variability in soil and rotational crops.

(e) References. The following references contain information for developing a protocol for study of the uptake of residues in rotational crops:

(1) Burnside, O.C. 1974. Trifluralin dissipation in soil following repeated annual applications. Weed Sci. 22:374-377. [The general methods used by this author could be adapted to small-scale field studies. The techniques used in this study should be considered for development of a protocol to obtain data on pesticide uptake with rotational crops.]

(2) Burnside, O.C., C.R. Fenster, and G.A. Wicks. 1971. Soil persistence of repeated annual applications of atrazine. Weed Sci. 19:290-293. [This paper contains procedures for investigation of pesticide fate with different soils and cropping practices.]

(3) Sirons, G.J., R. Frank, and T. Sawyer. 1973. Residues of atrazine, cyanazine, and their phytotoxic metabolites in a clay loam soil. J. Agr. Food Chem. 21:1016-1020. [This is not a study of uptake of a pesticide in a rotational crop system, but the procedures used could be applicable to design such a study. The study of a phytotoxic metabolite is included in this study.]

(4) [Reserved]

§ 165-3 Accumulation studies on irrigated crops.

(a) Purpose. The purpose of these studies is to determine residue uptake and levels in representative crops which are expected to be irrigated with water from a field treated with a pesticide (e.g., reclaimed waste water or water from a rice field used to irrigate upland crops). Such studies are needed to establish realistic label restrictions to prevent residues in irrigated crops and/or to provide information for a determination as to whether tolerances may be needed in the irrigated crop.

(b) When required. Data from a study of residue accumulation in irrigated crops under actual field use conditions are required by 40 CFR § 158 to support the registration of an end-use product intended for aquatic food crop or aquatic non-crop uses, for uses in and around holding ponds used for irrigation purposes, or for uses involving effluents or discharges to water used for crop irrigation. Data from such a study are also required to support each application for registration of a manufacturing-use product which legally could be used to make such an end-use product. See, specifically, 40 CFR § 158.50 and § 158.130 to determine whether these data must be submitted. Section II-A of this Subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(c) Test standards. Irrigated crop accumulation data submitted in response to 40 CFR § 158.130 should be derived from testing which complies with the general test standards in § 160-4 and all of the following specific test standards:

(1) Test substance. The test substance shall be a typical end-use product.

(i) If the applicant's product is an end-use product, the test substance shall be a product whose formulation is typical of the formulation category (e.g., wettable powder, emulsifiable concentrate, granular product) to which the product belongs.

(ii) If the applicant's product is a manufacturing-use product that legally could be used to make an end-use product for which irrigated crop accumulation data are required, the test substance shall be a product representative of the major formulation category which includes the end-use product. (If the manufacturing-use product is usually formulated into end-use products comprising two or more major formulation categories, a separate study must be performed with a typical end-use product for each such category.)

(2) Test procedures. (i) Field accumulation studies on irrigated crops should be conducted in at least two different sites which are representative of the areas where irrigated crops are expected to be grown.

(ii) Application. The irrigated crops should receive irrigation water containing the test substance at the highest expected concentration either consistent with the maximum rate recommended on the product label at the original site of application (aquatic food crop, aquatic non-crop, holding ponds) or consistent with the maximum rate calculated from direct discharges.

(iii) Irrigation. Irrigated crops should be irrigated at the maximum frequency consistent with good agricultural practices.

(iv) Sampling. (A) Foliage and/or crop produce samples should be collected from the test crops receiving irrigation water containing the test substance prior to the first irrigation treatment and at the earliest possible normal harvest interval following the last irrigation treatment.

(B) Water samples should be collected from the irrigation water at the time of each irrigation.

(C) Soil samples should be collected from the site of the irrigated crop prior to the first irrigation treatment, one day after each irrigation treatment, and at harvest of the irrigated crop.

(iv) Analysis. Residue data should be obtained for the soil, irrigation water, and irrigated crop samples collected during the course of this study.

(d) Reporting and evaluation of data. In addition to data submitted in response to the applicable reporting requirements specified in § 160-5, the test report should contain the following information:

(i) Analysis for residues of parent compound and degradates in the test crops, the irrigation water, and soil. Decline curves of the residues in soil should be included.

(ii) A description of residue data variability for the test crop, irrigation water, and soil sampled in this study.

(iii) Field test data, including:

(A) Dates of planting and harvesting;

(B) Method and frequency (times) of irrigation treatments;

(C) Sampling times and techniques;

(D) Stages of crop development at sampling times;

(E) Irrigation-to-harvest and irrigation-to-sampling intervals for each irrigation treatment;

(F) Depth, weight, or volume of each sample, and weights and volumes of aliquots taken for analysis;

(G) Flow data for irrigation water expressed in terms of volume or linear flow; and

(H) Rainfall data, temperature monitoring data, and a description of the general climatic conditions at the test sites.

(e) References. The following reference contains supplemental information for developing a protocol for conducting an accumulation study in irrigated crops:

(1) Demint, R.J., J.C. Pringle, Jr., A. Hattrup, V.F. Bruns, and P.A. Frank. 1975. Residues in crops irrigated with water containing trichloroacetic acid. J. Agr. Food Chem. 23:81-84. [This paper presents a procedure for assessment of pesticide carryover in irrigation water.]

(2) [Reserved.]

§ 165-4 Laboratory studies of pesticide accumulation in fish.

(a) Purpose. The purpose of these studies is to determine if pesticide residues accumulate in fish used as human food sources and to determine the extent of pesticide residues in edible portions of such fish. Data from pesticide accumulation studies in fish are used by the Agency to establish label restrictions (e.g., to prevent pesticide applications to certain sites so that there will be minimal residues entering edible fish or shellfish such as catfish or crayfish inhabiting rice fields). The data may also be used to

provide information for the setting of tolerances or action levels in these organisms where necessary.

(b) When required. (1) Data from a fish accumulation study are required by 40 CFR § 158 to support the registration of an end-use product intended for outdoor use (except domestic outdoor and greenhouse uses), or aquatic impact uses involving direct discharge of treated water into outdoor aquatic sites, except when any of the criteria in paragraph (b)(2) of this section are satisfied. Data from such a study are also required to support each application for registration of a manufacturing-use product that could be legally used to produce such an end-use product, except when any of the criteria in paragraph (b)(2) of this section are satisfied. See, specifically, 40 CFR § 158.50 and § 158.130 to determine whether these data must be submitted. Section II-A of this Subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(2) Fish accumulation data will not normally be required in situations where the registrant can offer evidence acceptable to the Agency showing that the active ingredient and/or its principal degradation product(s):

(i) Will not reach water, or

(ii) Will not persist in water (i.e., has a half-life of approximately four days or less), or

(iii) Has a relatively low potential for accumulation in fish as indicated by an octanol/water partition coefficient less than approximately 1000.

(c) Combined testing. Testing conducted as described in this section may be combined with testing conducted to meet the requirements of § 72-6 of Subdivision E, Hazard Evaluation: Wildlife and Aquatic Organisms, provided the test standards of each study are met.

(d) Test standards. Fish accumulation data submitted in response to 40 CFR § 158.130 should be derived from testing which complies with the general test standards in § 160-4 and all of the following specific test standards:

(1) Test substance. Accumulation studies shall be conducted using the active ingredient as the radioactively-tagged analytical grade or as the technical grade. If the study requires testing with a degradation product, the purest form that the applicant can obtain shall be used.

(2) Test procedures. (i) Residue studies should be conducted with radioisotopic (preferred) or non-radioisotopic analytical techniques.

(ii) Flow-through exposure studies are recommended. Exposure systems must maintain a constant concentration of chemical in a non-colloidal solution (use of carrier solvents to introduce the test substance to dilution water is permissible) and the concentration must not exceed 1/10 of the 96-hour LC_{50} of the test species. Within these constraints, the concentration of the test substance should be high enough to facilitate chemical identification of residues in fish. Bluegill sunfish or channel catfish are the preferred species, although other species may be appropriate. A control (non-treated) group of fish is recommended.

(iii) Exposure duration is 28 days with depuration (withdrawal) of 14 days with suggested sampling times and total residue analyses as follows:

(A) Water: days 0, 7, 14, 21, and 28 of the accumulation period;

(B) Fish: total residues in whole body, edible tissue, and viscera should be determined on days 0, 3, 7, 14, 21, and 28 of the accumulation period, and days 1, 3, 7, 10, and 14 of the depuration period.

(iv) Residues in two samples of edible tissue and two samples of viscera containing the highest residue levels during the accumulation period should be identified (if sufficient material exists). In general, extractable residues present at 0.05 ppm or greater should be identified.

(v) The Agency recognizes that special circumstances may be present (e.g., the occurrence of multiple persistent degradates, particular use patterns, or accumulation mechanisms) under which alternative experimental designs may be desirable. Therefore, the registration applicant may wish to discuss with the Agency the appropriateness of alternative experimental designs for fish accumulation studies (e.g., short-term kinetic studies or simulated field studies).

(e) Reporting and evaluation of data. In addition to the applicable reporting requirements specified in § 160-5, the following should be reported:

(1) A detailed description of the test conditions should be provided. This should include water characteristics (e.g., dissolved oxygen, pH, temperature, and dissolved salts), information on the test organisms (scientific name, source, weight, observed mortality, disease treatment, and acclimation procedures), and test methodology (detailed protocol, organism loading ratio, lighting, temperature, feeding schedule, and similar information).

(2) Total residue levels in whole body, edible tissue, and viscera, and in exposure water at all sampling times, including a description of data variability, shall be reported, as well as the identity of residues at specifed times.

(f) References. (1) The following reference is a review of accumulation of pesticides in fish:

Hamelink, J.L., and A. Spacie. 1977. Fish and chemicals: The process of accumulation. Ann. Rev. Pharmacol. Toxicol. 17:167-177. [This paper contains information on the parameters, kinetics, and processes that influence the accumulation of pesticides in fish. It could provide useful background information for design of accumulation studies.]

(2) The following references contain experimental procedures relative to design of studies of pesticide accumulation with sunfish:

(i) Branson, D.R., G.E. Blau, H.C. Alexander, and W.B. Neely. 1975. Bioconcentration of 2,2',4,4'-tetrachlorobiphenyl in rainbow trout as measured by an accelerated test. Trans. Am. Fish. Soc. 104:785792. [Accelerated accumulation tests such as those reported in this paper may be an acceptable substitute for full-length studies under certain conditions. Examples might include instances where concerns about multiple persistent degradates exist.]

(ii) Krzeminski, S.F., C.K. Brackett, and J.D. Fisher. 1975. Fate of microbicidal 3-isothiazolone compounds in the environment: Modes and rates of dissipation. J. Agr. Food Chem. 23:1060-1068. [This is an example of a flow-through study. It demonstrates high levels of steadystate accumulation under steady dosing conditions and a rapid residue decline when the dosing rate goes to zero.]

(iii) Macek, K.J., M.E. Barrows, R.F. Frasny, and B.H. Sleight, III. 1975. Bioconcentration of ^{14}C-pesticides by bluegill sunfish during continuous aqueous exposure. Pp. 119-142 in Structure Activity Correlations in Studies of Toxicity and Bioconcentration with Aquatic Organisms. G.D. Veith and D.E. Konasewich (eds). Great Lakes Advisory Board. International Joint Commission. Windsor, Ontario, Canada.

[This is a good prototype for design of a protocol for study of pesticide accumulation in fish.]

§ 165-5 Field accumulation studies of aquatic non-target organisms.

(a) Purpose. Field accumulation studies are required to determine if, following aquatic non-crop applications of pesticides, pesticide residues are accumulated in the edible tissues of fish that normally inhabit sites in or adjacent to these treated areas. The residue-bearing fish subsequently may be used as human food sources. Furthermore, these studies are needed to establish realistic label restrictions necessary to protect man and the environment and to provide information for determining if tolerances or action levels may be needed. These studies complement the laboratory data required for those uses by taking into account the contributions of pesticide degradation, partitioning, and movement under field use conditions in determining the amount and nature of the residues available to the nontarget aquatic organisms via accumulation.

(b) When required. (1) Data from a field accumulation study in aquatic nontarget organisms are required by 40 CFR § 158 to support the registration of an end-use product:

(i) Which is intended for forestry use, aquatic non-crop use, or aquatic impact use that results in direct discharges of treated water into outdoor aquatic sites;

(ii) For which data from the laboratory fish accumulation study (§165-4) show a potential for residues of the pesticide to accumulate.

(iii) For which no tolerance or action level for fish has been granted.

(2) Data from such a study are also required to support each application for registration of a manufacturing-use product which legally could be used to make an end-use product described in paragraph (b)(1) of this section. See, specifically, 40 CFR § 158.50 and § 158.130 to determine whether these data must be submitted. Section II-A of this Subdivision contains an additional discussion of the "Formulators' Exemption" and who must submit the required data as a general rule.

(c) Combined testing. Testing conducted as described in this section may be combined with testing conducted to meet the requirements of § 164-2 (field dissipation studies for aquatic uses and aquatic impact uses) or § 165-4 (laboratory studies of pesticide accumulation in fish), provided the test standards of each study are met.

(d) Test standards. Aquatic non-target field accumulation data submitted in response to 40 CFR § 158.130 should be derived from testing which complies with the general test standards in § 160-4 and all of the following specific test standards:

(1) Test substance. The test substance should be a typical end-use product representative of a major formulation category (e.g., wettable powder, emulsifiable concentrate, granular product) and containing the active ingredient of the applicant's product.

(i) If the applicant's product is an end-use product, the test substance should be a product whose formulation is typical of the formulation category to which the product belongs.

(ii) If the applicant's product is a manufacturing-use product that legally could be used to make an end-use product for which non-target organism field accumulation data are required. The test substance shall be a product representative of the major formulation category which includes that end-use product. (If the manufacturing-use product is usually formulated into end-use products comprising two or more major formulation categories, a separate study must be performed with a typical end-use product for each such category.)

(2) Test procedure. (i) The rate and method of test substance application should approximate the intended use pattern of the pesticide.

(ii) Fish (bottom, middle, and surface feeders, if available) including water, should be sampled in representative application areas.

(iii) Sampling times for fish and water should include pre-application, date of application, and immediate post-application for each single or multiple application of the test substance, and then at 3, 7, 14, 21, and 28 days following the last application.

(iv) Residue analyses should be performed on the whole body and edible tissue of fish and on the water samples for each sampling interval.

(e) Reporting and evaluation of data. In addition to the applicable reporting requirements specified in § 160-5, the reporting requirements specified in §§ 164-3(e) and 165-4(e) apply.

(f) References. (1) Consult the references cited in § 164-2(f) for guidance on the general conduct of aquatic non-target organism field accumulation studies.

(2) Consult the references cited in § 165-4(f) for guidance in the sampling and residue analysis of fish.